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User's Guide:
Chromeleon® Operational Qualification and Performance Qualification Software Templates for IC and BioLC Instruments, revision 5.11

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1 Introduction

Operational and Performance Qualification are becoming increasingly important to analytical laboratories. Documented evidence must be provided to demonstrate the integrity of data collected and qualify the results obtained on laboratory instrumentation.

The following institutes set and control industry instrumentation standards:

NIST. The National Institute of Standards and Technology is an administrative body of the US Department of Commerce. Together with the industry, the NIST develops new technologies and sets technological standards countrywide. Dionex includes NIST traceable standards as part of the qualification kit.

ASTM. The American Society for Testing and Materials sets technical standards and rules for the industry worldwide, specifically; E 1657 – 98: Standard Practice for Testing Variable-Wavelength Photometric Detectors in Liquid Chromatography. E1511 – 93: Standard Practice for Testing Conductivity Detectors used in Liquid Chromatography. E19.09.07: Proposed Standard Practice for Evaluation of Gradient HPLC pumps – Draft.

These standards are written in broad terms to make them as widely applicable as possible. All stipulate general requirements specifying instruments must be fit for purpose, technically checked in defined periods, calibrated and make sure that test results are documented according to international quality standards. The OQ/PQ procedure used to qualify Dionex Ion Chromatography Systems meets the requirements established by NIST and ASTM.

This OQ/PQ procedure provides qualification testing for **DX-500, DX-600, ICS-1000, ICS-1500, ICS-2000, ICS-2500, DX-320, DX-120, ICS-90, BioLC and ICS-3000** systems as well as any system containing components listed in section 1.5.

OQ/PQ results can be influenced by unusual laboratory conditions. For example, direct sunlight and close proximity to air conditioning ducts will cause thermal irregularities that will affect baseline drift on conductivity detectors. In these cases, it may happen that the manufacturer's specifications are not met, and the specifications may have to be adjusted accordingly.

All OQ/PQ activities must be formally recorded, all instruments must be clearly identified, and test reports and supporting documentation must include the following information:

- Module name, type and serial number
- Software and firmware version
- Test data
- Service interval
- Signature of the Dionex Service Engineer/Qualification Executor who performed the OQ/PQ
- Signature of the customer who reviewed and accepted the OQ/PQ results

1.1 How to use this manual

1. Determine which tests are required for the system to be qualified and write them down on a sheet of paper. This will help keep track of what steps to follow during system setup and preparation. It will also aid in selecting the appropriate sequences to run the tests.

2. For the DX500, DX600, and ICS2500 systems, electrochemistry cells are not used for any qualification tests apart from noise and drift qualification. If a customer uses amperometry methods, a conductivity cell must be used in its place to perform injector and gradient pump tests.

3. For the ICS-3000 system, electrochemistry cells in Integrated Amperometry mode are supported for qualification tests. If using Integrated Amperometry detection, a conductivity detector must be used in its place to perform a gradient pump test (pump flow test and EG test should also be run in conductivity mode). For a system with only electrochemical detectors, it is suggested that a complete system qualification be performed with a conductivity detector (including pump gradient accuracy, flow rate, and EG), after the system has been qualified with the conductivity detector, the detector should be exchanged for an ED detector. The ED tests (noise and drift, detector linearity) can be executed as described in this manual. The ICS-3000 electrochemistry detector DC amperometry and cyclic voltametry modes are not supported. When qualifying Integrated Amperometry detection a disposable gold working electrode is use. Other electrodes are not supported.

4. Use the **Table of Contents** and the **List of Tables** as reference guides to locate information quickly.

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1.3 Operational Qualification (OQ)

According to "The Development and application of guidance on equipment qualification of analytical instruments" by P. Bedson and M. Sargent [Accred. Qual. Assur. (1996) 1: 265 – 274] the following definition applies:

The purpose of OQ is to prove and document that an analytical system functions according to its operating specification while the specific environmental conditions are taken into account. In his specification, the supplier has therefore to define exactly the conditions that must be observed.

OQ should be performed after initial system installation or when an additional new module is added to an existing system. It is necessary that OQ be done according to the instructions outlined in this manual and all tests should be performed by a qualified Dionex Service Engineer.

1.4 Performance Qualification (PQ)

According to "The Development and application of guidance on equipment qualification of analytical instruments" by P. Bedson and M. Sargent [Accred. Qual. Assur. (1996) 1: 265 – 274] the following definition applies:

The purpose of PQ is to prove and document that an analytical system functions according to a specification that is suitable for the system's routine operation. A system is subject to wear when being operated. Thus, it may happen that the supplier's specification is no longer met. This means: The test procedures used for OQ are also used for PQ, but the tolerances of PQ are less restrictive than those for OQ. Using the same test procedures simplifies the handling.

PQ should be performed at regular intervals after the initial installation and OQ. Dionex recommends performing PQ every six months. It is necessary that PQ be done according to the instructions outlined in this manual and all tests should be performed by a qualified Dionex Service Engineer.

1.5 Basic Requirements for Successful OQ and PQ

It is a requirement that the most current revision of the OQ/PQ software templates is used in order for a qualification to be considered valid. It is the responsibility of the qualification engineer to determine the most up to date revision of the templates available and ensure that revision is used before proceeding with this qualification.

It is recommended that the qualification be performed using the most current Chromeleon service pack version (Contact Dionex Technical Support for current version).

It is recommended that the qualification be performed using the most current firmware version for each system or module (Contact Dionex Technical Support for current version).

As described in section 1.1, OQ and PQ are system-specific test procedures. The procedures described below are valid for IC systems comprising of the following equipment (only one type of detector and one pump can be qualified concurrently):

Pump: IP20, IP25, IS25, DX120, IC20, IC25, IC25A, GP40, GP50, GS50, ICS-90, ICS-1000, ICS-1500, ICS-2000, ICS-3000 SP, ICS-3000 DP.

Sampler: Manual injection: AS, AS40, AS50, TSP AS3500.

Absorbance detector: AD20, AD25, UVD-170U, PDA-100, UVD-340U, ICS Series VWD, ICS Series PDA.

Conductivity detector: CD20, CD25, CD25A, DX120, IC20, IC25, IC25A, ED40, ED50, ED50A, ICS-90, ICS-1000, ICS-1500, ICS-2000, ICS-3000 CD.

Electrochemical detector: ED40, ED50, ED50A, ICS-3000 ED.

Fluorescence detector: RF-2000 with UCI-50, UCI-100 or UI20 interface.

Eluent Generator: EG40, EG50, ICS-2000, ICS-3000 EG.

In addition, Chromeleon® version 6.5 or higher is required and the following requirements must also be met:

Module	Minimum Software Version
RF2000* with UI20	6.50 SP1
ICS-1000, ICS-1500, ICS-2000	6.50 SP2
AS50 (USB version)	6.60
AS	6.70
ICS-3000 SP or DP	6.70
ICS-3000 EG	6.70
ICS-3000 DC	6.70
ICS-3000 CD	6.70
ICS-3000 ED	6.70
FLUKE189 Multimeter	6.80
ICS Series VWD	6.80 SP1
ICS Series PDA	6.80 SP1
ICS-3000 TC	6.80 SP1

* RF-2000 with a UCI-100 or UCI-50 interface is supported with the Dionex Chromatography Management System version 6.4 or later.

It is possible to qualify modules in systems that include modules that are not listed in this section; however some modification of the included programs may be required. Such modifications are not within the scope of this document.

It is not possible to qualify multiple detectors on a single system, with the following exceptions: An RF-2000 detector must be qualified in series with a conductivity detector. Two ICS-1000, 1500 or 2000 systems can be qualified simultaneously when used as a simultaneous injection system with an AS50 (USB version) or AS autosampler; a 5000 or 10,000 µL injection syringe must be loaded onto the autosampler.

For the ICS-3000, it is not possible to qualify multiple pumps on a single system. If multiple pumps are on a single timebase, the second pump will be ignored by the programs.

Overview of the Tests

The following table provides an overview on the parameters to be tested and the limits recommended for OQ and PQ.

Instrument	Parameter	Description	Limits	
			OQ	PQ
All conductivity detectors. CD20, CD25, CD25A, ED40, ED50, ED50A, IC20, IC25, IC25A, DX-120, ICS-90, ICS-1000, ICS-1500, ICS-2000, ICS-3000 CD	Baseline Noise.	Pure water is pumped through the detector cell at 1.0 mL/min.	1 nS (DX-120 10 ns)	2 nS (DX-120 10 nS)
	Baseline Drift.		10 nS/h (DX-120 180 nS/h)	20 nS/h (DX-120 180 nS/h)
	Detector Linearity.	5 Nitrate solutions are injected in different concentrations. Concentration and peak height are represented on a graph. The coefficient of determination of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 5.0%	$r^2 \geq 0.999$ RSD ≤ 5.0%
AD20 and AD25, ICS Series VWD.	Baseline Noise.	Pure water is pumped through the detector at 1.0 mL/min. Wavelength 254 nm. Wavelength 520 nm.	AD20: 40 µAU AD25/VWD: 30 µAU	AD20: 50 µAU AD25/VWD 40 µAU
	Baseline Drift.		200 µAU/h	200 µAU/h
	Wavelength Accuracy.	Holmium oxide maxima are compared with the theoretical maxima.	AD20: ± 3 nm AD25/VWD ± 2 nm	AD20: ± 3 nm AD25/VWD ± 2 nm
	Detector Linearity.	5 Caffeine solutions are injected in different concentrations. Concentration and peak height are represented on a graph. The coefficient of determination of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 5.0%	$r^2 \geq 0.999$ RSD ≤ 5.0%

Instrument	Parameter	Description	Limits	
			OQ	PQ
PDA-100, ICS Series PDA	Baseline Noise.	Pure water is pumped through the detector at 1.0 mL/min. Wavelength 254 nm and 520 nm simultaneously.	0.04 mAU	0.10 mAU
	Baseline Drift.		0.5 mAU/h	1.0 mAU/h
	Wavelength Accuracy.	Holmium oxide maxima are compared with the theoretical maxima.	± 2 nm	± 2 nm
	Detector Linearity.	5 Caffeine solutions are injected in different concentrations. Concentration and peak height are represented on a graph. The regression coefficient of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 5.0%	$r^2 \geq 0.999$ RSD ≤ 5.0%
UVD-170U, UVD-340U	Baseline Noise.	Pure water is pumped through the detector at 1.0 mL/min. Wavelength 254 nm.	0.04 mAU	0.10 mAU
	Baseline Drift.		0.5 mAU/h	1.0 mAU/h
	Detector Linearity	5 Caffeine solutions are injected in different concentrations. Concentration and peak height are represented on a graph. The regression coefficient of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 5.0%	$r^2 \geq 0.999$ RSD ≤ 5.0%
Electro-chemical detectors. ED40, ED50, ED50A.	Baseline Noise.	An ED/CD Validation test cell is plugged into the Amperometry Cell connector. Potential: 1.0 V.	D.C Amp: 3 pA Int. Amp: 3 pC	D.C Amp: 4 pA Int. Amp: 4 pC
	Baseline Drift.		D.C Amp: 4 pA/h Int. Amp: 4 pC/h	D.C Amp: 8 pA/h Int. Amp: 5 pC/h
ICS-3000 ED	Baseline Noise.	50mM NaOH is pumped through the detector cell at 0.25 mL/min. A disposable gold working electrode is used running the Amino Acids (pH,Ag,AgCl Reference) waveform.	Int. Amp: 160 pC	Int. Amp: 160 pC
	Baseline Drift.		Int. Amp: 1250 pC/h	Int. Amp: 1250 pC

	Detector Linearity	5 Threonine solutions are injected in different concentrations. Concentration and peak height are represented on a graph. The regression coefficient of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 5.0%	$r^2 \geq 0.999$ RSD ≤ 5.0%

Instrument	Parameter	Description	Limits	
			OQ	PQ
Fluorescence Detectors. RF-2000	Baseline Noise.	Pure water is pumped through the detector cell at 1.0 mL/min. Excitation Wavelength 350 nm. Emission Wavelength is changed from 450 to 394 nm to give a response of between 40 and 80 mV. Noise is measured over 30 30-second intervals.	< 0.3 mV. Minimum Signal 40 mV. Maximum signal 80 mV.	< 0.3 mV. Minimum Signal 40 mV. Maximum signal 80 mV.
	Wavelength Accuracy	The emission wavelength is changed from 380 to 410 nm in steps of 1 nm every 15 seconds. The maximum of the emission spectrum is determined as the maximum of the signal.	≤ 10 nm	≤ 10 nm
All injectors and samplers.	Injector Precision.	10 injections at 25 µL each of the same standard are analyzed. The relative standard deviation of the peak areas indicate the reproducibility of the injection volume.	RSD ≤ 0.8%	RSD ≤ 1.0%
	Carry-over.	Following the injection of a highly concentrated test sample, a blank sample is injected, then a water blank sample is injected. The peak area measured in this chromatogram compared with the peak area of the highly concentrated sample indicates the amount of carry-over. If there is any peak area measued in the water blank sample, that area is subtracted from the area of the blank sample before it is compared to the highly concentrated sample.	≤ 0.1%	≤ 0.1%
AS, AS50 and TSP AS3500 Automated Sampler.	Linearity of Injector Volume.	5 injections of the same standard sample are analyzed. The injection volumes are different (10 µL to 50 µL). Injection volume and peak area are represented on a graph. The coefficient of determination of the resulting line and the deviations from it indicate the linearity.	$r^2 \geq 0.999$ RSD ≤ 1.0%	$r^2 \geq 0.999$ RSD ≤ 1.0%

	Sample Preparation Precision (if present – not supported on the TSP AS3500or AS with ICS-3000 ED detection).	3 samples of identical concentration are prepared from the same starting vial into 3 separate vials and injected. The relative standard deviation of the peak areas indicate the reproducibility of the sample preparation.	RSD ≤ 1.5%	RSD ≤ 2.0%

Instrument	Parameter	Description	Limits	
			OQ	PQ
EG40, EG50, ICS-2000, ICS-3000 EG.	Current Qualification	The eluent generator is commanded to produce a series of different concentrations (using the current cartridge type configured) (0 mM to 100 mM). At each different EG concentration setting the current produced by the eluent generator is measured and compared with theoretical values. For ICS systems with two EG cartridges linked to a single pump the eluent generator is commanded to produce a series of different concentration (using the current cartridge type configured) (0 mM to 50 mM) for each cartridge.	See report.	
ICS-3000 EG	Concentration reproducibility	The eluent generator is commanded to produce a series of different concentrations for KOH or MSA cartridge type. Each run is repeated 3 times and then evaluated for reproducibility. This test is only designed for KOH or MSA cartridges using an ICS-3000. This test is optional and is only part of the PQ and can only be run in conductivity mode.	N/A	≤ 0.5%
GP40, GP50, GS50, ICS-3000 SP, ICS-3000 DP.	Step Accuracy	A step gradient of channels A and B, then C and D is programmed and measured. Theoretical signal heights are compared with recorded signal heights for each step. The deviations between theoretical values and measured values indicate the step accuracy. For channel A and C water is used, for channel B and D 0.3% v/v acetone (absorbance detectors) or 1 mM KCl (conductivity detectors) is used.	≤ 2.0%	≤ 2.0%

	Step Noise	For each step of the gradient run described above the noise is determined relative to the signal height.	Microbore: ≤ 0.5% Standard bore or ICS-3000: ≤ 0.2%	Microbore: ≤ 0.5% Standard bore or ICS-3000: ≤ 0.2%
	Flow-Rate Accuracy	Water is pumped at 1.00 mL/min into a weighed beaker for exactly 5 minutes. The weight of the water is measured, the flow rate of the pump calculated and compared with the theoretical value. This experiment is repeated four more times.	0.05 mL/min	0.05 mL/min
	Flow-Rate Precision	Water is pumped at 1.00 mL/min into a weighed beaker for exactly 5 minutes. This procedure is repeated four more times. The relative standard deviation of the five measurements is calculated.	5.0%	5.0%
ICS-1000, ICS-1500 or ICS-2000	Column Heater Temperature Verification	The column heater is commanded to 40°C for the duration of the entire qualification. At the end of the qualification the temperature of the column heater is measured with a calibrated external thermometer and compared with the theoretical value.	± 2.0°C	± 2.5°C
ICS-3000 DC	Column TC and Compartment, Temperature Verification	The column/compartment heater is commanded to 30°C for the duration of the entire qualification. At the end of the qualification the temperature of the column heater is measured with a calibrated external thermometer and compared with the theoretical value.	± 2.0°C	± 2.5°C

Note: The instrument's specifications can only be met if the conditions that are described in the accompanying manual are strictly observed.

2 Qualification Preparation

2.1 Materials in Kit

Before starting the qualification procedure confirm that all the materials needed are present. If any items from these tables are not present, do not continue with the qualification process until these items are available.

Part Description	Part Number	Quantity
OQ/PQ User's Manual Version 5.11 (this document)	031726 – 15	1
Tubing, 0.010" (0.25 mm) ID black PEEK • *Connect pump to injection valve, backpressure regulator to detector, etc.*	042690	24", (81 cm)
25 µL Sample Loop • *AS40/Manual injection*	042857	1
100 µL Sample Loop • *AS/AS50*	042951	1
3 – Way mixing tees • *Connect eluent lines and eluent bottles*	024314	2
Fittings	043275	6
Ferrules	043276	6
Qualification stickers	050837	6
Certificate of Functional Performance	031423	6
NIST Traceable Caffeine Standards Kit • *Absorbance and Photodiode Array detectors*	060253	1
NIST Traceable Nitrate Standards Kit • *Conductivity detectors*	060254	1
Backpressure Regulator and Loop assembly (or equivalent) • *Generate system backpressure of 1400-1600 psi and delay volume for detector and injector tests*	2251.6010A	1
ED Validation Test Cell* **	049928	1
IC Validation/Test Box* ***	060615	1
EGC Sensing Plug***	063788	1
AD20 Holmium Oxide Test Cell* **	046172	1
Qualification Temperature Probe* ***	061329	1 or 2
EG40 Adaptor Cable* **	057899	1
OQ/PQ Qualification Templates 5.11 for CM 6.50 or later	057601 – 14	1
Test Binder	035863	1

* Included in the Dionex Chromatography Management System IC OQ/PQ Kit with Test Cells (Dionex P/N 057599) only. If using the Dionex Chromatography Management System IC OQ/PQ Basic/Refill Kit (Dionex P/N 057608), the Dionex Service Engineer must provide test cells and thermometer.

** These items are not needed for ICS-3000 system qualification and are not included in Dionex Chromatography Management System IC OQ/PQ Kit with Test Cells for ICS-3000 (Dionex P/N 063540).

*** Dionex Chromatography Management System IC OQ/PQ Kit with Test Cells for ICS-3000 (Dionex P/N 063540) contains two temperature probes for the dual oven configuration.

2.2 Additional Required Materials

The following items will be needed:

Description	Quantity
ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered) • *All eluent, standard, and sample preparation*	2L
Reagent Grade or better Potassium Chloride for OQ/PQ tests using a Conductivity Detector and a Gradient Pump • *Gradient pump tests using conductivity detectors*	1g
HPLC grade Acetone for OQ/PQ Tests using an Absorbance or Photodiode Array Detector and a Gradient Pump • *Gradient pump tests using absorbance and photodiode array detectors*	3mL
Sodium Hydroxide 50/50 w/w (Low Carbonate) (Dionex Part Number 033465) • *Eluent for Integrated Amperometry qualification on and ICS-3000*	~5.2mL
Traceable Threonine Standards • *Integrated Amperometry qualification on and ICS-3000***	1
Disposable Amino Acid Gold Working Electrode (Dionex Part Number 060082) • *Integrated Amperometry qualification on and ICS-3000****	1
CarboPac PA1 guard 2mm (Dionex Part Number 057179) • *Integrated Amperometry qualification on and ICS-3000****	1
Ammeter or multimeter with milliamp (mA) setting for OQ/PQ tests using an eluent generator. Must be capable of displaying 0.00 to 220 mA • *Eluent Generator current test*	1
Dionex conductivity cell (detector) • *Detector, injector and gradient pump tests using conductivity detectors in conductivity mode* • *Detector linearity, Injector and gradient pump tests using an DX500, DX600, ICS-2500 ED detector in conductivity mode normally used in an amperometry mode* • *Gradient pump tests using an ICS-3000 normally used in an ED mode*	1
100 mL volumetric flasks (Class A, 100 ± 0.08 mL) • *Standard preparation for AS40 and Manual Injection*	5
1000 mL volumetric flasks (Class A, 1000 ± 0.3 mL) • *Eluent preparation*	At least 1
Transfer Pipettes or Pipettor	0.5 – 10.0 mL
Calibrated Analytical Balance • *Weigh KCl for eluent preparation* • *Weigh eluents for the FlowRate Accuracy and Precision test.*	0.000g
Computer workstation running Chromeleon™ or PeakNet™ Version 6.3 or later (later versions required for some configurations, see Section 1.5)	1

****** These items are needed for ICS-3000 system qualification in ED mode. These items are included in the ICS-3000 ED Qualification kit (Dionex part number 063541)**

2.3 Preparation of Standards

Two standards kits are provided in the OQ/PQ kit. Use the Caffeine kit, Part Number 060253 to qualify systems with an absorbance detector (AD20, AD25, UVD-170U, PDA-100 or UVD-340U). Use the Nitrate kit, Part Number 060254 to qualify systems with a conductivity detector.

To qualify an ICS-3000 system with an ED detector in Integrated Amperometry mode use the Threonine standard kit for ED qualification (Dionex P/N 063541). ED detectors qualification does not support AS40 auto samplers.

2.3.1 Caffeine standard preparation for the Injector Precision test (AS40 autosamplers only).

(AS40 Autosamplers only) For the Injector Precision test prepare a 40 ppm caffeine standard using the 1000 ppm caffeine standard ampoule included in the Caffeine Standards Kit. Pipette 4.0 mL of the 1000 ppm caffeine standard into a 100 mL volumetric flask. Fill the flask to the mark with ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered).

(AS40 Autosamplers and Manual Injection) For the Carry Over test prepare a 250 ppm caffeine standard using the 1000 ppm caffeine standard ampoule included in the Caffeine Standards Kit. Pipette 2.5 mL of the 1000 ppm caffeine standard into a 10 mL volumetric flask. Fill the flask to the mark with ASTM Type 1 (or better) deionized water (10 MΩ-cm, filtered).

2.3.2 Nitrate standard preparation for the Injector Precision test (AS40 autosamplers only).

For the Injector Precision test prepare a 50 ppm nitrate standard using the 1000 ppm nitrate standard ampoule included in the Nitrate Standards Kit. Pipette 5.0 mL of the 1000 ppm nitrate standard into a 100 mL volumetric flask. Fill the flask to the mark with ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered).

For the Carry Over test use the included 1000 ppm nitrate standard neat.

2.4 Preparation of Eluents

All eluents must be prepared using ASTM Type 1 (or better) deionized water (18 MΩ, filtered).

2.4.1 Preparation of Eluents for use with Conductivity Detection

These eluents are used for qualification with an ED40/ED50/ED50A, CD20/CD25/CD25A, IC20/IC25/IC25A, DX-120, ICS-90, ICS-1000, ICS-1500, ICS-2000 or ICS-3000 Conductivity Detector.

Prepare the following eluents and connect them to lines A, B, C and D of the pump. All eluents must be degassed. Eluent bottles must be pressurized when using a GP40, IP20, IC20, DX-120 or ICS-90 pump. Eluent lines A and C can be connected to one eluent bottle containing 1 L of water by a mixing tee. Eluent lines B and D can be connected to one eluent bottle containing 1 L of 1 mM KCl by a mixing tee.

Note: Eluents B, C and D are needed for the Gradient Pump Accuracy Test only and should be prepared only if the pump configured has gradient capabilities. Do not prepare these eluents if the

pump configured is an IP20/IP25, IC20/IC25/IC25A, IS25, DX-120, ICS-90, ICS-1000, ICS-1500, ICS-2000 or ICS-3000 Isocratic pump.

Eluent	Description
A	Deionized water
B	1.0 mM KCl
C	Deionized water
D	1.0 mM KCl

10 mM KCl Stock Solution Preparation:

Prepare one liter of 10 mM KCl by weighing 0.746 grams of KCl. Place the KCl in a 1L volumetric flask. Fill flask to the mark with ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered) water.

1 mM KCl Preparation:

Pipette 100 mL of the 10 mM KCl solution into another 1L volumetric flask, filling the flask to the mark with ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered) water. The 1 mM KCL solution should be freshly prepared daily.

Remember to …

- Degas all eluents.
- Connect eluent lines A and C to one eluent bottle containing 1 L of deionized water equipped with a 3-way mixing tee.
- Connect eluent lines B and D to one eluent bottle containing 1 L of 1 mM KCl equipped with a 3-way mixing tee.
- Pressurize eluent bottles with GP40/IP20/IC20/DX-120 pumps.
- Eluent bottles on other pumps do not need to be pressurized.

2.4.2 Preparation of Eluents for use with Absorbance Detection

These eluents are for used for qualification with an AD20, AD25 or UVD-170U Absorbance Detector or the UVD-340U or PDA-100 Photodiode Array Detector.

Prepare the following eluents and connect them to lines A, B, C and D of the pump. All eluents must be degassed. Eluent bottles must be pressurized when using a GP40, IP20, IC20, DX-120 or ICS-90 pump. Eluent lines A and C can be inserted into one eluent bottle containing 1 L of water. Eluent lines B and D can be inserted into one bottle containing 1 L of 0.3% Acetone in Water.

Note: Eluents B, C and D are needed for the Gradient Pump Accuracy Test only and should be prepared only if the pump configured has gradient capabilities. Do not prepare these eluents if the pump configured is an IP20/IP25, IC20/IC25/IC25A, IS25, DX-120, ICS-90, ICS-1000 ICS-1500, ICS-2000 or ICS-3000 Isocratic pump.

Eluent	Description
A	Deionized water
B	0.3% Acetone in Water
C	Deionized water

D	0.3% Acetone in Water

0.3% Acetone in Water Preparation:

Pipette 3 mLs of HPLC grade Acetone into a 1L volumetric flask. Fill flask to the mark with ASTM Type 1 (or better) deionized water (18 MΩ-cm, filtered) water. Eluents should be freshly prepared on a daily basis.

Remember to …

- Degas all eluents.
- Connect eluent lines A and C to one eluent bottle containing 1 L of deionized water using a 3-way mixing tee.
- Connect eluent lines B and D to one eluent bottle containing 1 L of 1 mM KCl using a 3-way mixing tee.
- Pressurize eluent bottles with GP40/IP20/IC20/DX-120 pumps.
- Eluent bottles on other pumps do not need to be pressurized.

2.4.3 Preparation of Eluent for use with Integrated Amperometry Detection

This two different eluent is used for qualification with an ICS-3000 Electrochemical Detector in Integrated Amperometry mode.

For Flow Test (initial Flow test):

Eluent	Description
A	Deionized water

For all other ED Tests (will be used in section 3.3.2 step 18c):

Eluent	Description
A	50 mM NaOH

The preparation of 50 mM NaOH for ED tests should be performed after the FLOW test has been completed to ensure minimal potential contaimination of NaOH eluent. Do not make the 50 mM NaOH eluent until section 3.3.2 step 18c. Prepare the following eluent and connect them to line A of the pump. All eluents must be degassed.. Eluent lines A should be connected to one eluent bottle containing 1 L of 50 mM NaOH . After the 50 mM NaOH has been connected to line A, the line should be thoroughly primed.

50 mM NaOH Preparation:

Fill and eluent bottle with 997.4 mL of ASTM Type 1 (or better) deionized, degassed water (18 MΩ-cm, filtered) water. Pipette 2.6 mL of 50/50 (w/w) NaOH solution into the bottle. Cap, cover with He head pressure and mix. The 50 mM NaOH solution should be freshly prepared daily and care should be taken minimize exposure to CO_2 gas.

Remember to …

- Degas all eluents.

- Connect eluent line A to one eluent bottle containing 1 L of deionized 50 mM NaOH.
- Pressurize eluent bottles with He gas.

2.5 Preparing the IC System

Note: The AD25, VWD, PDA, and PDA-100 Detector UV and visible lamps must be turned on at least two hours prior to performing the qualification. AD20 Detector UV and visible lamps must be turned on to the high setting for at least 24 hours before performing the qualification. The UVD-170U and UVD-340U UV lamps must be turned on at least two hours prior to performing the qualification.

DX500, DX600, and ICS-2500 Systems equipped with an amperometry detector proceed directly to Section 2.5.1.

1. Open the PeakNet or Chromeleon Server Configuration application. Only one pump, one injection valve and one detector can be qualified concurrently (except the RF2000 which must be qualified in series with a conductivity detector), so if more than one pump, one injection valve or one detector are installed, the second pump (Pump_2), the second detector (CDet_2, EDet_2) will be ignored if both detectors are the same. If the detectors are different, uncheck the detector signal that will not be qualified. If the second pump or detector is to be qualified, remove (or uncheck) the first pump or detector in the timebase. If qualifying an ICS-1000, ICS-1500, ICS-2000, ICS-3000, DX-320, DX-120 or ICS-90 system, proceed directly to step 5. Make note of any changes made to the system for the purposes of this qualification. After qualification these changes should be reversed. It is helpful to save a backup file of the configuration before you make any changes for qualification.

2. PUMP (EXCEPT FOR ICS-3000 PUMP): Double click on the pump to be qualified to view the properties page for the pump. Select the **General** page and confirm the Device Name is "Pump" . Select the **Head Type and Limits** page and confirm the Head Type matches the heads installed on the pump. Select the **EluentGenerator** page (if installed) and confirm the Device Name is "EluentGenerator". Ensure the Offset Volume is set to 400.0 µL or less. Exit the properties page.

3. ELUENT GENERATOR: If using Chromeleon 6.60 or greater with a stand-alone eluent generator driver, double click on the eluent generator to be qualified to view the properties page for the eluent generator. Select the **General** page and confirm the Device Name is "EluentGenerator". On the Pump Link & Cartridge page confirm the eluent generator is linked to the pump to be qualified and the Offset Volume is set to no more than 400.0 µL.

4. DETECTOR (EXCEPT FOR ICS-3000 DETECTOR): Double click on the detector to be qualified to view the properties page for that detector. Select the **General** page and confirm the Device Name is "UV" for AD20, AD25, PDA-100, UVD-170U and UVD-340U detectors, or "ECD" for CD20/25/25A and ED40/50/50A detectors. Select the **Signals** page and confirm that the signal factor for absorbance, conductivity, fluorescence and DC amperometry detectors is 1.000. For integrated amperometry detectors confirm the signal factor is 1000.0. If it is not, change the factor to the correct value for the qualification. Confirm the signal unit is µS, nC or µA for conductivity, integrated amperometry or DC amperometry respectively. Confirm the signal unit is AU for AD20 and AD25 detectors and mAU for PDA-100, UVD-170U and UVD-340U detectors. Confirm the signal unit is mV for fluorescence detectors. If it is not, change the unit to the correct value for the qualification. For ECD systems select the **Heat Source** page and confirm the correct heat source is selected. Exit the properties page.

5. AUTOSAMPLER: Double click on the Autosampler to be qualified (if installed) to view the properties page for the autosampler. Select the **General** page and confirm the Device Name is "Sampler". Select the **Options** page and confirm the options match the configuration of your

system. Select the **Temperature** page and confirm the Autosampler controlled Column Temperature option matches your system (if installed). Exit the properties page. If validating a DX-500, DX-600 or Bio-LC system, proceed directly to step 16.

6. ICS-2000, ICS-1500 or ICS-1000: Double click on the ICS system to be qualified to view the properties page for the system. Select the **General** page and confirm the Device Name is "Pump_ECD". Select the **Options** page and confirm the Installed Options match the configuration of the system. If a degas is installed, ensure it is not in Cycle mode for the duration of the qualification. Select the **Signals** page and confirm the ECD_1 signal is active, the unit is µS and the factor is set to 1.000.

7. ICS-2000: Select the **EluentGenerator** page and confirm the EGC-1 cartridge is installed, the Device Name is "EluentGenerator" and a valid eluent generator cartridge serial number is entered.

8. ICS-3000 Pump: Double click on the pump to be qualified to view the properties page for the pump. Select the **General** page and confirm the Device Name is "Pump_1" or "Pump_2".

9. ICS-3000 DC: Double click on the DC to be qualified to view the general properties. Select the Thermal Controls page and confirm the heating configuration of the timebase. Confirm that the device name for the Column TC is "Column_TC" and the device name for the Compartment TC is "Compartment_TC" if installed on the timebase.

10. ICS-3000 DETECTOR: Double click on the detector to be qualified to view the properties page for that detector (for CD and ED this will be in the DC under the detector tab). Select the **General** page and confirm the Device Name is "UV" for AD20, AD25, PDA-100, UVD-170U and UVD-340U detectors. For the ED and CD detectors: Open the properties of the DC and click on the **Detectors** page. Highlight the detector name and click on **Configuration** page. Confirm that the Device Name is "CDet1" for CD detectors or "EDet1" for ED detectors. If both detectors are on the same timebase, uncheck the detector(s) that is not being qualified (only one detector can be qualified at a time). Click on the **Signals** page. Confirm the signal name is "CD_1" for CD detectors or "ED_1" for ED detectors. Confirm that the signal factor for absorbance, conductivity, fluorescence and Integrated amperometry detectors is 1.000. For integrated amperometry detectors confirm the signal factor is 0.001. If it is not, change the factor to the correct value for the qualification. Confirm the signal unit is µS, or nC for conductivity or integrated amperometry respectively. Confirm the signal unit is AU for AD20 and AD25 detectors and mAU for PDA-100, UVD-170U and UVD-340U detectors. Confirm the signal unit is mV for fluorescence detectors. If it is not, change the unit to the correct value for the qualification. Exit the properties page.

11. ICS-3000 ELUENT GENERATOR: Double click on the eluent generator to be qualified to view the properties page for the eluent generator. Select the **General** page and confirm the Device Name is "EluentGenerator". On the "cartridge" tab confirm the eluent generator is linked to the pump to be qualified. Confirm that there is either a serial number for a KOH or a MSA cartridge. If a serial number is not available for KOH or MSA cartridge, contact Dionex Technical Support. **At this point connect the EG test box with Ammeter to the EGC_1 connector. If there are two EGC devices on the same timebase connect the EGC sensing plug (Dionex part number 063788) connector (or a second EG test box with Ammeter) to the EGC_2 connector. If a second Ammeter is not available, use a jumper to connect the "EG internal Load" to the "COM" connection on the second test box, failure to do this will result in "over voltage" errors during the test**.

12. DX-320: Double click on the IC system to be qualified to view the properties page for the system. Select the **General** page and confirm the Device Name is "Pump_ECD". Select the **Head Type and Limits** page and confirm the Head Type matches the heads installed on the pump. Select the **EluentGenerator** page (if installed) and confirm the Device Name is

"EluentGenerator". Ensure the Offset Volume is set to 400.0 µL or less. Exit the properties page and proceed directly to step.

13. DX-120: Double click on the DX-120 system to be qualified (DX-120 systems only) to view the properties page for the system. Select the **General** page and confirm the Device Name is "Pump_ECD". Exit the properties page. **Note:** The default Device Name for a DX-120 system is "ECD"; the qualification will not work correctly unless it is renamed to "Pump_ECD". An updated panel is provided on the OQ/PQ CD to allow control of a DX-120 with Device Name "Pump_ECD".

14. ICS-90: Autosamplers other than the AS40 are not supported. Double click on the ICS-90 to be qualified. Select the General page and confirm the Device Name is Pump_ECD. Select the Signals page and confirm the signal name is set to ECD_1 and the factor is 1.00000 µS.

15. If applicable make sure the detector Output is in 'Offset' mode in the Analog Out Set-Up screen on the front panel.

16. Make note of any changes and save the configuration.

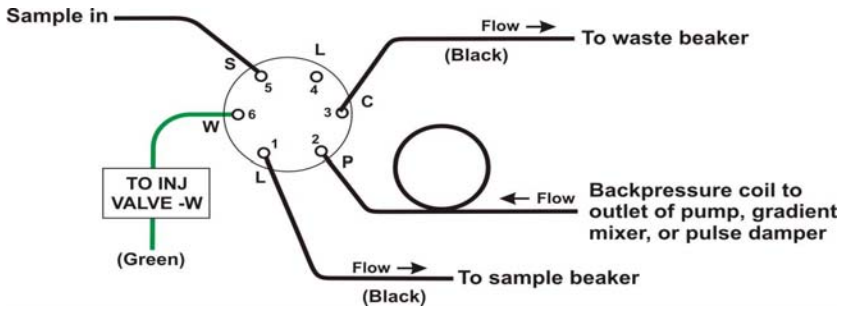
17. All modules, with the exception of the TSP AS3500, DX-120, AD20, RF2000 and AS40, must have their serial number set in the moduleware. Qualification of modules without serial numbers, with the exception of the above-mentioned modules, is not supported.

18. Connect Eluents A, B, C and D to the pump and prime each eluent line individually for approximately 3 minutes each, longer if the vacuum degas option is installed (10 minutes). Only connect Eluent A if the pump configured is an ICS-3000, ICS-2000, ICS-1500, ICS-1000, IP20/IP25, IC20/IC25, IS25, DX-120 or ICS-90 Isocratic pump. It is always recommended to pressurize the eluent bottles, but the eluent bottles must be pressurized when using GP40, IP20, IC20, DX-120 and ICS-90 pumps.

19. Connect the pump line to injection valve port 2 (P). Bypass the column, suppressor, CR-TC, and CRD from the system leaving the gradient mixer in-line; use the backpressure coil to connect the pump outlet directly to port 2 on the injection valve. If an Eluent Generator Cartridge is in-line with the IC system, bypass all plumbing to the EGC and connect the pump directly to the injection valve.

    a. ICS-2000: Disconnect the Pulse Damper Out line (0.030" tubing exiting from left and below the detector cell) and connect it directly to the Injection Valve port 2 (P), thus bypassing the eluent generator.

    b. Diagram on how to connect the pump outlet to the valve



20. ICS-2000, ICS-1500 and ICS-1000 (WITH COLUMN HEATER INSTALLED): Open and remove the column heater plastic cover by removing the two thumbscrews. Remove the column heater front plate by removing the six thumbscrews. Unclip and remove the column and insert the Robo-Thermometer in its place so that the LCD display sits out the top of the column heater with the probe lowered into the column clips. Replace the column heater front plate and the plastic

cover, leaving the thermometer LCD display out of the top of the cover and the probe in the column heater assembly. The LCD display of the thermometer should be visible and display the current column heater plate temperature. (Column heater plate temperature may vary slightly from the column heater liquid temperature).

21. ICS-3000 DC COLUMN TC: Horizontally insert the Robo-Thermometer in the 2nd tubing slot from the bottom of the DC so that the LCD display sits out the side of the column TC. The LCD display of the thermometer should be visible and display the current Column TC temperature.

22. ICS-3000 DC COMPARTMENT TC: Horizontally insert the Robo-Thermometer in top tubing slot on the DC so that the LCD display sits out the side of the column TC. The LCD display of the thermometer should be visible and display the current Column TC temperature.

23. ICS-3000 TC: Horizontally insert the Robo-Thermometer into the side tubing slot on the TC so that the LCD display sits out the side of the TC. The LCD display of the thermometer should be visible and display the current TC temperature.

24. RF-2000 DETECTOR: Make sure the fluorescence detector is plumbed in series with a conductivity detector (ED or CD detector). The RF-2000 detector will be qualified during the other tests. The tests will not be performed correctly if the RF-2000 detector is the only detector found on the system.

25. Thoroughly rinse the autosampler fluidics with water. This can be accomplished on the AS, AS50 and TSP AS3500 by flushing the sample syringe two or three times and on the AS40 by running two or three rinse vials.

26. AS, AS50 AND TSP AS3500: For systems using an AS, AS50 or TSP AS3500 Autosampler, install the 100 µL sample loop on the injection valve. From the front panel of the AS50, go to the **Main Menu**, select **Module Set-Up** and then **Plumbing Configuration**. If necessary change the sample loop size to 100 µL. See the users manual for the TSP AS3500 for instructions on how to install and configure the system for a 100 µL sample loop.

27. AS40: For systems using an AS40 Autosampler or manual injector, install the 25 µL sample loop on the injection valve. Connect the AS40 **Load** connector to **Relay_1** on integrated systems or to **Relay_1** on the pump for modular systems.

    a. Note: All programs using the AS40 are designed to run with the 5.0 mL vials (i.e. trigger relay at -2.40 minutes). If 0.5 mL vials are to be used programs will have to be manually modified to trigger the relay at -1.00 minute).

28. ICS-90 AND DX-120: Check that the flow rate of the pump is 1.0 mL/min. Adjust the flow rate accordingly until a flow rate of 1.0 mL/min is achieved. Let the system equilibrate for a minimum of 15 minutes. Monitor the detector output for a stable response.

29. ALL PUMPS OTHER THAN THE ICS-90 AND DX-120: Select 100% of Eluent A (water) and a flow rate of 1.00 mL/min. Turn the pump flow on and let the system equilibrate for a minimum of 15 minutes. If a degas is present ensure it is not in cycle mode.

30. AS40: If using an AS40 Autosampler, connect the 'LOAD IN' relay on the rear panel of the AS40 to RLY 1 OUT on the pump. For DX-120 and ICS-90 systems with an AS40 Autosampler connect the 'LOAD IN' relay on the rear panel of the AS40 to TTL 1 OUT on the DX-120 or ICS-90.

31. NEW TUBING: When running a qualification on a system with new tubing, such as a new backpressure coil, new sample loop or a complete new system, it is recommended to rinse the tubing with carbonate eluent before proceeding with the qualification. This can be achieved by injecting two or three times with 0.5 M Sodium Carbonate solution while running DI water at 1.00 mL/min, or by running 9 mM Sodium Carbonate as the eluent for 10 minutes before rinsing

with DI water for 20 minutes. The latter is recommended for new systems being qualified for the first time. ICS-1000, 1500 and 2000 systems ship with pre-rinsed tubing, it is not necessary to carry out this step with these systems.

32. CONDUCTIVITY OR ABSORBANCE: Pipette the standards from the standards kit ampoules directly into autosampler vials and place them in the position / order according to the table below. For the Injector Precision and Carry Over standards (AS40 Autosampler only) transfer the Injector Precision standards prepared in Section 2.3 into six autosampler vials and the Carry Over standard (Absorbance detectors only) into one autosampler vial. To qualify an IC system without an autosampler, use the AS40 table for the injection order and inject the samples directly from the standards kit ampoules.

33. ICS-3000 INTEGRATED AMPEROMETRY: Cut the label off the vials (included in the ICS-3000 ED qualification kit) and place the vial directly into autosampler in the position order according to the table below. Note that even though the main portion of the label has been cut off, the concentration marker is still adhered to the label.

34. For systems using an EG40, EG50, ICS-2000, ICS-3000 EG:

    a. Disconnect the Eluent Generator Cartridge cable from the Eluent Generator EGC connector.

    b. If installed, disconnect the CR-TC cable from the CR-TC connector.

    c. Plug the EG/AES/SRS Supply Input cable (4-pin connector) on the IC Validation/Test Box into the Eluent Generator EGC connector. EG40 users will need to use an EG40 Adaptor Cable (P/N 057899).

       Note: This test requires the IC Validation/Test Box. EG Validation Test Cells are no longer supported for OQ/PQ qualifications.

    d. Turn on the ammeter or multimeter and set the display to mA. This test requires mA readings from 0.00 to 220 mA.

    e. Connect the positive lead of the ammeter to the "EG Internal Load" red post connector on the IC Validation/Test Box.

    f. Connect the negative lead of the ammeter to the "COM" black post connector on the IC Validation/Test Box.

    g. For an ICS-3000 with two EGCs configured on one timebase, a EGC sensing plug (Dionex part number 063788)**or** a second Validation test box with Ammeter (see steps c through f) should be plugged into EGC_2. Failure to make a connection to EGC_2 this will result in "Over Voltage" errors when running the test. The program **XQ_EG_DUAL_CURRENT** or **XQ_ED_EG_DUAL_CURRENT** will prompt to switch the EGC_1 and EGC_2 during the run.

    h. From the Browser in the Dionex Chromatography Management System, go to the Panel directory and select the pump_EluGen_detector panel appropriate for the system being qualified. Go to Control and select the timebase configured with the Eluent Generator to be qualified (for Chromeleon 6.7, click on the "Default Panel Tab set".

    i. From the Eluent Generator section of the panel, select the cartridge type to be used. The panel will recognize previously configured KOH and MSA cartridges even though the IC Validation/Test Box is plugged into the cartridge connector.

35. FLUKE 189 MULTIMETER: Install the Fluke 189 multimeter into the timebase to be qualified. The driver for the Fluke 189 multimeter is included with Chromeleon 6.8 or later. Connect the Fluke 189 multimeter to the IC Validation/Test Box as described in step 33 of this section. The

Fluke 189 multimeter is only used for systems that require EG current qualification. It is recommended that the "Power off time out" for the Fluke189 be set to a value greater then the time it will take to perform the Operational Qualification. See Fluke189 operator's manual for instructions on how to set this value.

IMPORTANT NOTES:

a. It is important that the Fluke 189 multimeter is installed AND connected before loading the OQ/PQ templates

b. Follow the instructions in the Chromeleon 6.8+ help topics for the installation of the Fluke 189 multimeter Windows USB driver and for the Chromeleon device driver for the Fluke 189 multimeter

c. Follow the Fluke 189 multimeter instructions for adjusting the default 'PrOFF' setting in the meter. It is recommended to adjust this parameter to a longer time (*e.g.*, several hours or more) to avoid having the meter auto power off during your OQ/PQ tests.

AS/ AS50 / TSP AS3500 Autosampler Sample Order

Conductivity Detector with AS/AS50	Absorbance or Photodiode Array Detector with AS/AS50	ICS-3000 Integrated Amperometry Detector with AS	Vial Tray Position for AS orAS50 / TSP AS3500
Water	Water	Water	1 / A1
5 ppm Nitrate Std	-----	-----	2 / A2
10 ppm Nitrate Std	-----	-----	3 / A3
25 ppm Nitrate Std	-----	-----	4 / A4
50 ppm Nitrate Std	-----	-----	5 / A5
100 ppm Nitrate Std	-----	-----	6 / A6
1000 ppm Nitrate Std	-----	-----	7 / A7
Empty Vial*	-----	-----	8 / A8
Empty Vial*	-----	-----	9 / A9
Empty Vial*	-----	-----	10 / A10
-----	15 ppm Caffeine Std	-----	11 / A11
-----	40 ppm Caffeine Std	-----	12 / A12
-----	60 ppm Caffeine Std	-----	13 / A13
-----	80 ppm Caffeine Std	-----	14 / A14
-----	100 ppm Caffeine Std	-----	15 / A15
-----	1000 ppm Caffeine Std	-----	16 / A16
-----	Empty Vial*	-----	17 / A17
-----	Empty Vial*	-----	18 / A18
-----	Empty Vial*	-----	19 / A19
-----	-----	-----	20
-----	-----	1 uM Threonine Std	21
-----	-----	2 uM Threonine Std	22
-----	-----	4 uM Threonine Std	23
-----	-----	8 uM Threonine Std	24
-----	-----	10 uM Threonine Std	25
-----	-----	10 uM Threonine Std	26

- Vials only need to be present if the detector listed at the top of the table is to be qualified

* Needed only if qualifying AS50 Sample Prep. Option. AS50 10 mL vials are required for Sample Prep. Qualification.

AS40 Autosampler or Manual Injection Sample Order (Conductivity or Absorbance Only)

Qualification	Conductivity Detector with AS40	Absorbance or Photodiode Array Detector with AS40	Sample Order
Warm Up	Water	Water	Vial 1
Injector Precision	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 2
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 3
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 4
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 5
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 6
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 7
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 8
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 9
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 10
	50 ppm Nitrate Std*	40 ppm Caffeine Std*	Vial 11
Carry Over	1000 ppm Nitrate Std	250 ppm Caffeine Std*	Vial 12
	Water	Water	Vial 13
	Water	Water	Vial 14
Detector Linearity	5 ppm Nitrate Std	15 ppm Caffeine Std	Vial 15
	10 ppm Nitrate Std	40 ppm Caffeine Std	Vial 16
	25 ppm Nitrate Std	60 ppm Caffeine Std	Vial 17
	50 ppm Nitrate Std	80 ppm Caffeine Std	Vial 18
	100 ppm Nitrate Std	100 ppm Caffeine Std	Vial 19

* AS40 users: Do not use the standard from the standards kit for the Injector Precision and Absorbance Carry Over samples; instead, use the standards prepared in Section 2.3 of this manual.

2.5.1 DX500, DX600 and ICS-2500 Systems equipped with an Amperometry Detector

1. Open the PeakNet or Chromeleon Server Configuration application.
2. Double click on the detector to be qualified to view the properties page for that detector. Select the **General** page and confirm the Device Name is "ECD". Select the **Signals** page and confirm that the signal factor is 1.000. For integrated amperometry detectors confirm the signal factor is 1000.0. If it is not, change the factor to the correct value for the qualification. Exit the properties page.
3. Make note of any changes and save the configuration.

4. Disconnect the Amperometry cell cable from the ED40/ED50/ED50A Detector.

5. Plug the ED/CD Validation Test Cell Amperometry cable into the ED40/ED50 Amperometry Cell connector.

6. From the front panel of the detector, turn the cell on.

7. Let the detector equilibrate with the validation cell for approximately five minutes.

8. Proceed to 2.6 and finish the qualification. After qualifying the Noise and Drift, remove the ED/CD Validation Test Cell and install a Conductivity Cell. Change the mode of the detector to conductivity and restart the qualification process to qualify the rest of the system.

2.6 OQ/PQ Sequence Installation

The following are the required steps to perform the OQ/PQ with the Dionex Chromatography Management System:

Template sequences are generated based on the master sequence of the OQ/PQ CD (P/N 057601) and copied for each test that is performed on the same system. Instruments OQ/PQ are performed by processing the sequences of the copy. In this way, the template sequences must be adjusted only once to the IC configuration to be tested.

The sequences required for OQ/PQ of a particular IC configuration are available on the OQ/PQ CD in the IC_XQ_Main directory. To install the sequences required for your system, proceed as follows:

1. If using a sequential AS autosampler, manually put the diverter valve in the correct position for the timebase.

2. Open the "**Qualification**" menu in the Browser.

3. Select "**OQ Setup**" or "**PQ Setup**". A wizard opens and assists you in copying the sequences. Use the "Next >" and "< Back" buttons to navigate the steps of the wizard.

4. Select the timebase for which you wish to perform OQ or PQ and enter the computer name where it is installed. Click "Next >".

5. Make sure the OQ/PQ CD is in the CD drive. Select "Get the files from the CD" making sure the drive letter selected is the drive with the OQ/PQ CD. Click "Next >".

6. The Dionex Chromatography Management System will automatically create a default unique name and directory to contain all template sequences, which is suitable for most circumstances. Change the name or directory if required. Click "Next >".

7. A list of sequences is displayed. These sequences will be copied to the corresponding datasource. Click "Finish".

8. When the installation is complete the "Specification" page opens in the report. The Print Setup dialog may open to confirm your paper size and printer destination. Select OK. If asked to use the new settings for all sheets of the current printer layout, select Yes.

9. Remove the write protection (click on "Layout Mode" in the "Edit" menu) and make the following entries:
   - customer name, customer company, qualification executor name, qualification executor company.
   - period between qualifications (default is 6 months)
   - note: PeakNet and Chromeleon version 6.30 build 587 incorrectly report build 576 to the

report, manually correct this error if applicable.
Instrument names and test limits will be filled in automatically into the report if it is opened after the "Warm Up" sample is run. During the copying this is not the case. Please do **not** change the appropriate cells in the Instruments and Limits sections.

10. Activate the write protection (click "Layout Mode" in the "Edit" menu).
11. Save the report ("Workspace" menu – "Save Report Definition"). Click "Yes" if asked to overwrite an existing file.
12. If performing an OQ on 2 channels sequentially (shared AS), repeat all steps for the second timebase

The program files of the installed sequences apply to the following instruments, instrument names and channel names as determined in the PeakNet or Chromeleon Server Configuration Program:

Instrument	Name
IP20/25, IS25, GP40/50, GS50	Pump
ICS-3000 Pump	Pump_1, Pump_2
AD20/25, VWD, PDA, PDA-100, UVD-170U, UVD-340U	UV
RF2000	Emission
UV channel names	UV_VIS_1, UV_VIS_2
CD20/25/25A, ED40/50/50A	ECD
ECD channel name	ECD_1
Amperometry channel names (tri-mode drivers only)	IntAmp_1, DCAmp_1
ICS-3000 Conductivity Detector	CDet1
ICS-3000 Conductivity channel name	CD_1
Fluorescence channel name	Emission
ICS-3000 Conductivity channel name	EDet1
ICS-3000 Conductivity channel name	ED_1
AS, AS50, AS3500	Sampler
EG40, EG50, ICS-2000, ICS-3000 EG	EluentGenerator
ICS-2000, ICS-1500, ICS-1000, IC20/25/25A, DX-120, ICS-90	Pump_ECD

If there are modules in the system that do not match the above names, automatic selection of the tests to perform and the corresponding sequences will not be possible.

2.7 OQ/PQ Sequence Execution

Note: For ICS-3000 Integrated Amperometry System: Before executing the sequences, first turn on the flow and the cell. Click on the "Default Panel Tabset". From the pump tab, start the pump flowing 50 mM NaOH at 0.25 mL/min. Click on the EC detector tab, Select Cell mode of "IntAmp", then click on the "Waveform" button, use the drop down menu to choose the waveform "Amino Acids (pH,Ag,AgCl Reference)), then click on the "Exit" button. Turn the cell voltage to "On".

1. Open the "**Qualification**" menu in the Browser.

2. Select "**Instruments OQ**" or "**Instruments PQ**". A wizard opens and assists you in copying the sequences. Use the "Next >" and "< Back" buttons to navigate the steps of the wizard.

3. Select the timebase and computer name of the system for which you want to perform OQ or PQ. Click "Next >".

4. Select the source directory of the template to be used (this is the directory chosen in Section 2.6, step 6 and normally will be chosen automatically). Click "Next >".

5. The Dionex Chromatography Management System will automatically create a default unique name and directory to contain the copy, which is suitable for most circumstances (default: template name + date). Change the name or directory if required. Click "Next >".

6. A list of sequences is displayed containing all sequences of the corresponding template. Click on the sequences required for the tests.
   A copy is made and the batch list of the respective timebase is automatically updated and opened. Click on Ready Check to confirm the program and method files contained in the sequence are compatible with the configured system. Make modifications to the program files if necessary.
   Note: If an Eluent Generator is configured on the system, the following message may occur at the Ready Check: "Flow rate not specified at time 0.0, using a flow setting 1.00." This refers to the Eluent Generator flow rate, not the pump flow rate. It is not necessary to take any action since the Eluent Generator is not used for this part of the qualification. Proceed with starting the batch.
   If qualifying an AD20 absorbance detector, close the Start Batch dialog box by clicking on the Cancel button. Otherwise, start the batch to process the sequences.

   Note: For systems with a shared auto sampler there might be a number of informational messages indicating that "Exclusive Access" has not be acquired or released. This is normal and occurs because the qualification does not release the auto sampler until the qualification is complete.

Sequence Name	Check
XQ_Flow_Rate	Pump flow-rate accuracy and precision tests for all pumps
XQ_IC_Flow_Rate	Pump flow-rate accuracy and precision tests for all pumps excluding the ICS-90 and DX-120.
XQ_EG_Current	Current qualification of an EG, EG40 or EG50 Eluent Generator
XQ_EG_Dual_Current	Current qualification of an Dual EG configuration (ICS-3000 with both EGC configured on one timebase)
XQ_IC_EG_Current	Current qualification of an EG40, EG50, (DX-320 IC systems only) or ICS-2000 Eluent Generator
XQ_Warm_Up	Warm-up sequence for all systems (except ICS-3000 with ED detector)
XQ_Wavelength_Accuracy	Wavelength accuracy test for AD25 and PDA-100 absorbance detectors
XQ_CD_Noise_Drift	Noise and drift test for conductivity detectors
XQ_DC_Amp_Noise_Drift	Noise and drift test for DC amperometry detectors (ED single-mode driver DX500, DX600 or ICS-2500)
XQ_DCAmp_Noise_Drift	Noise and drift test for DC amperometry detectors (PN/CM 6.40+ tri-mode driver DX500, DX600 or ICS-2500)
XQ_IC_Noise_Drift	Noise and drift test for ICS-90, ICS-1000, ICS-1500, ICS-2000, DX-320 and DX-120 IC systems
XQ_Int_Amp_Noise_Drift	Noise and drift test for integrated amperometry detectors (ED single-mode driver DX500, DX600 or ICS-2500)
XQ_IntAmp_Noise_Drift	Noise and drift test for integrated amperometry detectors (PN/CM 6.40+ tri-mode driver DX500, DX600 or ICS-2500)
XQ_PDA_Noise_Drift	Noise and drift test for photodiode array detectors
XQ_UV_Vis_Noise_Drift	Noise and drift test for absorbance detectors (excluding PDA detectors)
XQ_Abs_Inj_Precision	Injector precision test for systems with an absorbance detector
XQ_Abs_Inj_Carry_Over	Injector carryover tests for systems with an absorbance detector
XQ_CD_Inj_Precision	Injector precision test for systems with an electrochemical detector
XQ_CD_Inj_Carry_Over	Injector carryover test for systems with an electrochemical detector
XQ_IC_Inj_Precision	Injector precision test for DX-320 and DX-120 systems
XQ_IC_Inj_Carry_Over	Injector carryover test for DX-320 and DX-120 systems
XQ_ED_Warm_Up	Warm-up sequence for ICS-3000 systems with ED detector

XQ_ED_Noise_Drift	Noise and drift test for ICS-3000 amperometry detectors (ED in Integrated Amperometry mode)
XQ_ED_Inj_Precision	Injector precision for ICS-3000 systems with amperometry detector (ED in Integrated Amperometry mode)
XQ_ED_Carry_Over	Injector carry over for ICS-3000 systems with amperometry detector (ED in Integrated Amperometry mode)
XQ_ED_Linearity	Detector Linearity for ICS-3000 systems with amperometry detector (ED in Integrated Amperometry mode)
XQ_ED_Inj_Linearity	Injector linearity for ICS-3000 systems with amperometry detector (ED in Integrated Amperometry mode)
XQ_ED_Stop	The sequence turns off the pump and cell for ICS-3000 systems with amperometry detector (ED in Integrated Amperometry mode).
XQ_Abs_Linearity	Detector linearity test for absorbance detectors
XQ_CD_Linearity	Detector linearity test for electrochemical detectors
XQ_IC_Linearity	Detector linearity test for ICS-90, ICS-1000, ICS-1500, ICS-2000, DX-320 and DX-120 IC systems
XQ_Abs_Inj_Linearity	Injector linearity test for systems with an absorbance detector
XQ_CD_Inj_Linearity	Injector linearity test for systems with an electrochemical detector
XQ_IC_Inj_Linearity	Injector linearity test for ICS-90, ICS-1000, ICS-1500, ICS-2000, DX-320 and DX-120 systems
XQ_Abs_Samp_Prep	Injector sample preparation precision test for systems with an absorbance detector
XQ_CD_Samp_Prep	Injector sample preparation precision test for systems with an electrochemical detector
XQ_IC_Samp_Prep	Injector sample preparation precision test for ICS-90, ICS-1000, ICS-1500, ICS-2000, DX-320 and DX-120 systems
XQ_Fluor_Wavelength_Noise	Fluorescence detector noise and wavelength accuracy tests
XQ_GP_Grad_Accuracy	Gradient accuracy and noise test for systems with a gradient pump
XQ_Stop	The sequence slows down the flow rate to 0.25 mL/min to conserve eluent for overnight runs
XQ_IC_Stop	The sequence stops the flow to conserve eluent for overnight runs

7. The OQ/PQ qualification tests are performed in the following order:

   a. Wavelength Accuracy (only if an AD20 absorbance detector is present).

   b. Flow-Rate Accuracy and Precision test (all pumps with the exception of the ICS-90 and DX-120).

c. Eluent Generator Current Qualification Test (only if an EG40, EG50 or ICS-2000 is present).

d. Warm Up.

e. Wavelength Accuracy (only if an AD25 or PDA-100 absorbance detector is present).

f. Detector Noise and Drift Test.

g. Injector Precision Test.

h. Carry Over Test.

i. Detector Linearity Test.

j. Injector Linearity Test (only if an AS, AS50 or AS3500 is present).

k. Sample Preparation Precision Test (only if an AS, AS50 is present with the Sample Prep Option and the 10 mL Tray Type installed).

l. Fluorescence Detector Noise Test (only if an RF2000 detector is present).

m. Fluorescence Detector Wavelength Accuracy Test (only if an RF2000 detector is present).

n. Gradient Accuracy Test (only if a gradient pump is present).

8. Test 'a' is done manually and does not require a sequence template. The program will pause before the Eluent Generator Current Qualification Test (only if an EG40, EG50 or ICS-2000 is present) and the Flow-Rate Accuracy and Precision Test as these tests require user intervention.

2.7.1 Evaluating the Test Sequences

The master sequence on the OQ/PQ CD and all copies made from it for OQ and PQ are linked to the corresponding report. **Do not change this report** (exceptions; step 9, Section 2.6 and below). In the report, many references are used between the individual data sheets. When lines or columns are deleted or inserted, those references may be lost so the calculations may be wrong!

Select the XQ_Warm_Up sequence and open the Water_WU sample in the Dionex Chromatography Management System browser. Open the Printer Layout (View menu) and select the Specification page from the tabs at the bottom of the report. Make sure the report is in Layout Mode (Edit menu). If the Moduleware Versions are not recorded, enter Moduleware Versions into the Moduleware Version column of the Instruments section as well as any Descriptions, Lot and Serial Numbers and Expiration Dates in the accessories section. ***Note:*** Leading zeros will be truncated by the Chromeleon report unless an inverted comma (') is entered before the serial or lot number. Turn off Layout Mode and save the sheet (Save Report Definition… in the Workspace menu or just close the report and click Yes when asked if Chromeleon should save the changes).

Select the XQ_ECD_Linearity, XQ_Abs_Linearity, XQ_ED_Linearity or XQ_IC_Linearity sequence and open the ECD_Linearity.qnt, UV_Linearity.qnt, ED_Linearity.qnt or IC_Linearity.qnt method file. Open the Amount Table by clicking on the Amount Table tab at the bottom of the screen. The Amount Table should contain five amount columns, Amount Detector linearity_1 through 5, not necessarily in order. For ECD and IC systems the columns will contain amount values for the nitrate standards, for Abs systems it will contain amount values for caffeine standards and for ICS-3000 ED systems it will contain amount values for threonine standards. These values should be modified to match the exact reported amounts listed on the certificate of analysis that came with your standards kit. For example, if the reported amount for the 5 ppm nitrate standard is 4.97 ppm, the value in the Amount Detector linearity_1 column should be changed to 4.97. Save (Save command in the File menu) and close the method file.

To ensure that the measured values are correctly read and processed in the report it **must** be printed as "Batch Report" from the browser. Select the sequence for which to print the report, ensure that no sample is marked, select "Batch Report" in the "File" menu and start printing by clicking "ok". It is possible to highlight all completed sequences and print all the reports at once.

2.7.2 Repeating Individual Tests

It may be necessary to repeat individual tests. If so, review Section 5 where problems are described according to which individual tests might fail. According to Good Laboratory Practice, all tests following the one that failed must be repeated. The entire OQ/PQ qualification is designed such that almost all tests require that the previous one be passed successfully.

For example, if the test regarding the linearity of a UV detector fails, the results regarding the linearity of the injector are questionable as the linearity of the detector is a prerequisite for testing the injector linearity.

3 Performing the Qualification

3.1 OQ/PQ Test Order

The OQ/PQ qualification tests are performed in the following order:

a. Wavelength Accuracy Test (only for AD20 Absorbance Detectors).

b. Flow Rate Accuracy and Precision Qualification.

c. Eluent Generator Current Qualification Test (only for EG40, EG50, ICS-2000 and ICS-3000 eluent generators).

d. Warm Up.

e. Wavelength Accuracy Test (only for AD25, VWD, PDA and PDA-100 Absorbance Detectors).

f. Detector Noise and Drift Test.

g. Injector Precision Test.

h. Carry Over Test.

i. Detector Linearity Test.

j. Injector Linearity Test (only for AS, AS50 and TSP AS3500 autosamplers).

k. Sample Preparation Precision Test (only for AS and AS50 autosamplers with the Sample Prep Option and 10 mL Tray Type installed).

l. Fluorescence Detector Noise Test (only if an RF2000 detector is present).

m. Fluorescence Detector Wavelength Accuracy Test (only if an RF2000 detector is present).

n. Gradient Accuracy Test (only for gradient pumps).

o. Column Heater Temperature Verification (only for ICS-1000 with column heater option, ICS-1500 and ICS-2000 systems).

p. Column TC and/or Compartment TC (only for the ICS-3000)

q. EGC reproducibility (optional as a separate test for PQ only)

3.2 OQ/PQ Test Timetable

Test	Method	Material Required	Equilibration Time	Approx. Run Time
Wavelength Accuracy AD20	Manual	Test Cell	Lamps – 24 hrs 10 mins w/test cell	Up to 10 mins
Flow Rate Accuracy and Precision All pump models except ICS-90 and DX-120.	Sequence	- Eluent (water) - Backpressure loop	15 mins	30 mins
Wavelength Accuracy AD25, ICS Series VWD	Sequence	Eluent (water)	Lamps – 2 hrs 10 mins w/pump	6 mins
Wavelength Accuracy PDA-100, ICS Series PDA	Sequence	Eluent (water)	Lamps – 2 hrs 10 mins w/pump	6 mins
DC Amperometry Noise / Drift	Sequence	Test cell	5 mins	20 mins
Int. Amperometry Noise / Drift	Sequence	Test cell	5 mins	20 mins
Fluorescence Noise and Wavelength Accuracy	Sequence	Eluent (water)	Lamps – 2 hrs 10 mins w/pump	25 mins
AS40 / Manual Inj Warm Up Detector Noise / Drift Injector Precision Carry Over Detector Linearity	Sequence	- Eluent (water) - Standards - Backpressure loop	10 minutes	1hr, 10 mins
AS/ AS50 / TSP AS3500 Warm Up Detector Noise / Drift Injector Precision Carry Over Detector Linearity Injector Linearity	Sequence	- Eluent (water) - Standards - Backpressure loop	10 minutes	2 hr, 10 mins
AS / AS50 with Samp Prep Warm Up Detector Noise / Drift Injector Precision Carry Over Detector Linearity Injector Linearity Sample Prep Precision	Sequence	- Eluent (water) - Standards - Backpressure loop	10 minutes	2 hr, 40 min
Eluent Generator	Sequence	- Test cell	N/A	16 mins

Current Qualification		- Multimeter		
Gradient Accuracy GP40 and GP50 microbore	Sequence	- Eluents - Backpressure loop	10 mins	62 mins
Gradient Accuracy ICS-3000 SP/DP, GS50 and GP50 standard bore	Sequence	- Eluents - Backpressure loop)	10 mins	43 mins
Column Heater, TC, Column TC, Compartment TC Temperature Verification	Manual	Robo-Thermometer	15 mins minimum	0 min
EGC reproducibility	Sequence (PQ)	-DI Water -Backpressure EGC cartridge	1 hours	3 hours

3.3 Starting the Sequences

<u>For AS Autosamplers with the Sequential Injection Option</u>: Before starting any sequences, first verify that none of the timebases configured on the computer have "Acquired Exclusive Access" to the autosampler, or column TC, or compartment TC. If this is the case release the "Exclusive Access" command before proceeding. Once the qualification sequences begin, the system being qualified will gain and retain exclusive access to the autosampler, column TC and compartment TC until the "Stop" sequence is run and "Exclusive Access" is released. "Exclusive Access" can be released by going to the command function (F8), choosing the module from the list, and executing the "ReleaseExclusiveAccess" command.

The wavelength accuracy test for AD20 absorbance detectors is the only tests performed manually. The wavelength accuracy test for AD20 absorbance detectors must be completed before starting the main qualification. Proceed directly to section 3.3.4 if using a detector other than the AD20.

3.3.1 Wavelength Accuracy (AD20 Absorbance Detector only)

1. Remove the AD20 Sample cell from the photodiode and disconnect the beam-splitter cable.
2. Install the AD20 Holmium Oxide Test cell (Dionex P/N 046172) onto the photodiode and connect the beam-splitter cable.
3. Make sure the UV and Visible lamps are set to HI. Set the detector wavelength to 354 nm and then press **Enter** on the front panel. Allow the detector to equilibrate with the test cell installed for at least 10 minutes before performing the Wavelength Accuracy test.
4. Copy the **OQ_UV.rdf** or **PQ_UV.rdf** report file, depending on the qualification being performed, from the PN_CD datasource under the PQ_OQ and Templates directories to the folder for the OQ runs. Locate the row of sheet names at the bottom of the report. Click on the **DET_AD20_WAVE** sheet. Print the sheet. This will allow for manual recording of test results. Test results can also be directly entered into the report (see Step 10). Close the report.

Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

5. From the front panel of the AD20 detector, press the **Offset** button to zero the detector reading. Allow the detector to equilibrate for approximately 20 seconds. Record the offset absorbance value as the measured absorbance for 354 nm. This value should be approximately 0.0000 AU.

6. Manually change the wavelength setting on the front panel of the AD20 to 355 nm and press **Enter** on the front panel. Allow the detector to equilibrate for approximately 20 seconds. Record the resulting absorbance value for 355 nm.

7. Repeat Step 6 for the remaining 13 wavelength settings in the **DET_AD20_WAVE** sheet.

8. After all wavelength absorbance values have been recorded, find the wavelength setting that produced the maximum absorbance value.

9. Record the wavelength setting that produced the maximum absorbance value.

10. Open the **OQ_UV.rdf** or **PQ_UV.rdf** report file from the location it was copied to in step 4 (do not open the file from the CD as it cannot be modified). Go to **Edit** and turn **Layout Mode** on. Update the **SPECIFICATION** page with the correct information. Go to the **DET_AD20_WAVE** sheet. Enter today's date in the **Additional Information** section. Enter all previously recorded values in the table on the sheet. In some cases a cell value may need to be modified (i.e. change the detector serial number), for this modification Chromeleon Report Publisher is required. If Chromeleon Report Publisher is not available, the cell can be modified by clearing the value of the cell and typing in the new value. The deviation and pass/fail results will be calculated automatically. Print the sheet. Click on **Edit** and turn **Layout Mode** off. Save the report file to the local datasource by selecting Save Report Definition… from the Workspace directory. Choose the same datasource and directory used for the main part of the qualification.

11. If any part of the test does not pass, refer to Section 5 for troubleshooting assistance.

3.3.2 Flow Rate Accuracy and Precision Qualification

The Flow Rate Accuracy and Precision test is designed to qualify the flow-rate accuracy and precision of all pumps, with the exception of the ICS-90 and DX-120. No flow-rate test is required for ICS-90 and DX-120 systems.

Systems equipped with an AS3500 autosampler cannot be qualified automatically by this procedure. Dionex does however suggest the use of alternative protocols to verify flow rate measurement. Such tests should be able to determine flow rate accuracy and precision in accordance with the specifications outlined on page 10. Dionex cannot guarantee tests performed outside the scope of the published OQ/PQ protocol.

1. Ensure the pump is disconnected from the Dionex Chromatography Management System. The system should be in hold mode and a message box displaying "Plumb pump for Pump Flow-rate Accuracy and Precision Test, press OK to continue" should be visible.

2. Stop the pump from the front control panel of the module if it is not already stopped. ICS-1000 users: if the pump is not already stopped, it may be necessary to click OK on the message box, manually stop the batch and turn off the pump from the control panel. Remember to reset the status to single before resuming the test.

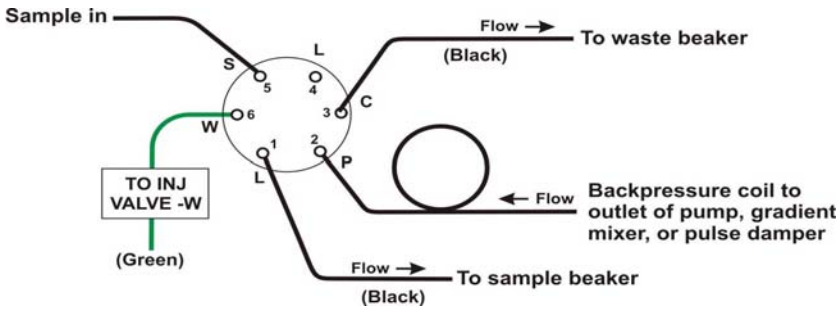
3. Locate the backpressure coil.

4. Connect one end of the backpressure coil directly to the outlet of the pump, after any pulse dampener loops or gradient mixers if applicable and the other to port 2 (P) of the injector valve.

5. Prepare two lengths of black tubing, 0.010 (0.25mm) ID, of equal length, approximately 12" (40 cm). Connect one of these lines to port 1 (L) of the injector valve. The tubing connected to port 1 (L) of the injector valve will be the eluent line that goes to the "sample beaker.".

6. Connect the other line to port 3 (C) of the injector valve. The tubing connected to port 3 (C) of the injector valve will be the waste line.

7. Run the waste line to a waste beaker, and the eluent line to a sample beaker. The plumbing to the valve should look like the figure below.



8. Turn the pump on (except ICS-1000 or ICS-3000 users).

9. Click OK on the Dionex Chromatography Management System. The system should reconnect to the module and/or resume the program in run mode. ICS-1000 and ICS-3000 users: the pump should turn on at this point.

10. The program will equilibrate the pump for 15 minutes before starting the test. During this time eluent flow should pass through the waste line. Ensure that sufficient backpressure is observed during this equilibration time.

11. At time 0, the injector will switch to the eluent line.

12. At time 2.00, the injector will switch back to the waste line and the Dionex Chromatography Management System will ask you to "Please weigh the eluent container. Enter results under segment 0. Press OK to continue." The program will be in hold mode and will wait until OK is pressed before continuing. Weigh the eluent container, but do not zero the balance used for weighing. Record the weight as segment 0. Click OK to continue.

    Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

13. The Dionex Chromatography Management System will switch the injector back to the eluent line for exactly 5 minutes. After this time the injector will switch to the waste line and a message "Please weigh the eluent container. Enter results under segment 1. Press OK to continue" will be displayed. The program will be in hold mode and will wait until OK is pressed before continuing. Weigh the eluent container, but do not zero the balance used for weighing. Record the weight as segment 1. Click OK to continue.

14. Repeat 12 for segments 2 through 5.

15. After the sequence is finished open the Water_FR sample from the browser. Click on Printer Layout from the View Menu. Select the Flow Rate tab at the bottom of the page, turn on layout mode and enter the Segment values and room temperature into the sheet. Save the worksheet by selecting Save Report Definition… from the Workspace menu.

16. Print the Flow Rate Accuracy and Precision report by selecting Print… from the File menu. Choose a printer and press OK to begin printing.

17. If any part of the test does not pass, make the appropriate repair or calibrations and rerun the test. Refer to Section 5 for troubleshooting assistance.

18. Plumb the system for the remainder of the qualification.

19. Ensure the pump is disconnected from the Dionex Chromatography Management System. The system should be in hold mode and a message box displaying "Plumb the system for the remainder of the qualification tests, press OK to continue" should be visible.

    a. All Configurations Except ICS-3000: Connect the regulator end of the Backpressure Regulator and Loop Assembly (P/N 2251.6010A) to port three (C) of the injection valve, using a small length (approx. 15 cm) of 0.010" tubing. Attach the loop end to the inlet of the conductivity or absorbance cell, thus bypassing all columns and suppressors.
    It is important to connect the Backpressure Regulator and Loop assembly with the regulator upstream from the loop or unusual peak shapes may occur.

        i. **Note: If the Backpressure Regulator and Loop Assembly (P/N 2251.6010A) is unavailable, a piece of 0.003" tubing may be used to provide approximately 1500 psi (± 200 psi) backpressure and sufficient delay. thus bypassing the eluent generator.**

    b. ICS-3000 Conductivity or Absorbance: Connect the regulator end of the Backpressure Regulator and Loop Assembly (P/N 2251.6010A) to the heat exchanger outlet line. Attach the loop end to the inlet of the conductivity or absorbance cell, thus bypassing all columns and suppressors.
    It is important to connect the Backpressure Regulator and Loop assembly with the regulator upstream from the loop or unusual peak shapes may occur.

        i. **Note: If the Backpressure Regulator and Loop Assembly (P/N 2251.6010A) is unavailable, a piece of 0.003" tubing may be used to provide approximately 1500 psi (± 200psi) backpressure and sufficient delay.**

    c. ICS-3000 Integrated Amperometry: Calibrate the reference electrode (see operators manual), then assembly the ED cell with an AAA certified disposable gold working electrode (P/N 060082) and mount the ED cell onto the ED detector. Connect the two cell leads (yellow and blue) to the appropriate ports on the detector. Connect a 24-inch piece of 0.003" PEEK tubing to port three (C) of the injection valve, connect the other end to the inlet of a CarboPac PA1 guard (2-mm) column. To the outlet of the guard column connect a small length (approx 15-cm) of 0.010" tubing. Attach the loop end to the inlet of the Integrated Amperometry Cell.

        i. **Note: When loading the Theronine standards in the AS autosampler, it may be necessary to cut the label off the Threonine vial for proper fit into the AS sample tray. For this reason, the concentration has been printed on the part of the label that will remain on the vial.**

3.3.3 Eluent Generator Current Qualification

The Eluent Generator Current Qualification test (j) can be used for an EG40, EG50, ICS-2000 or ICS-3000 EG (single or dual) configured with either a KOH or MSA cartridge (or both).

1. **Resume** the Batch by clicking OK. This sequence is an Eluent Generator step gradient. Each step is held until the user clicks OK to continue. There may be a delay of up to 0.4 minutes

between when the Eluent Generator program steps to the next concentration and when the ammeter or multimeter registers the new milliamp reading. Record the milliamp reading for each step on a note pad. These values will be entered into the **EG_CURRENT, EG_DUAL_CURRENT** or **EluGen Current Qualification** sheet after the run is complete. The run time is 11.0 minutes plus pause time.

Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

2. When the run is complete, open the report file by double clicking on the Water_EG sample of the XQ_EG_Current sequence and select Printer Layout from the View menu. Locate the row of sheet names at the bottom of the report and click on the **EG_CURRENT, EG_DUAL_CURRENT** or **EluGen Current Qualification** sheet.

3. Go to **Edit** and turn **Layout Mode** on. Enter the milliamp readings that were recorded in step 1 for each concentration value in the **EG_CURRENT, EG_DUAL_CURRENT** or **EluGen Current Qualification** sheet. The deviation and pass/fail results will be calculated automatically.

4. Print the EluGen Current Qualification report by selecting Print… from the File menu. Choose a printer and press OK to begin printing.

5. If any part of the test does not pass, make the appropriate repair/adjustments and rerun the test. Refer to Section 5 for troubleshooting assistance.

6. To qualify another cartridge, select the new cartridge type in the control panel, add the XQ_EG_Current, XQ_EG_Dual_Current or XQ_IC_EG_Current sequence to the batch list, set the status of the Water_EG and Stop samples to single and repeat steps 1 - 4.

3.3.4 Performing the Main Qualification

The main part of the OQ/PQ consists of the tests d – n, as shown in Section 3.1. This part of the qualification requires that an absorbance or conductivity detector be configured on the system being qualified. For DX500, DX600 and ICS 2500 systems using an amperometry detector, only test f applies.

If the qualification will be performed using a manual injection valve, use full loop injections and overfill the loop a minimum volume equal to of 3 times the sample loop volume. Inject standards at the beginning of each sample in the sequence. There will be a 2.5-minute delay before the sample is injected. It is advisable to manually edit the programs to replace the 2.5-minute delay with a message command to hold the system until the sample is ready. Replace the "`-2.500 Pump_Relay_1.Closed Duration=130.00`" line with "`-0.100 Message "Load the sample into the injection loop, press OK to continue"`"

Note: AD20 and AD25 sequences contain both 254 nm and 520 nm Noise and Drift Tests. If you do not want to perform the Noise and Drift tests at both wavelengths, change the sequence status line of the test you do not want to run to **Finished,** then save the sequence. The PDA-100 sequence collects data at both wavelengths simultaneously. The UVD-170U and UVD-340U sequences only collect data at 254 nm.

1. The IC must be set up and running as described in Section 2.5.

2. The sequences must be installed as described in Section 2.6.

3. The sequences must be set up as described in Section 2.7.

4. If the batch is not started already go to Batch and Edit. Start the Batch. Run times are as follows:

Systems with an AS40 or manual injection have a run time of approximately 1hr, 10 min.
Systems with an AS, AS50 or TSP AS3500 but no sample prep have a run time of approximately 2hr, 10 min.
Systems with an AS, AS50 and sample prep have a run time of approximately 2hr, 40 min.
Systems with a gradient pump, add an extra 60 minutes to the above times.
At the end of the main qualification the system will automatically slow down to 0.25 mL/min to conserve eluent for overnight runs. For ICS-3000, ICS-2000, ICS-1500, ICS-1000, DX-320, DX-120 and ICS-90 systems the flow will stop.
For systems using an Eluent Generator, the program will pause before the Eluent Generator Current Qualification Test, proceed to section 3.3.3. The program will pause before the Flow-Rate Accuracy and Precision Test, proceed to section 3.3.4 before continuing to step 5.

5. When the Batch is finished, go to the Dionex Chromatography Management System Browser and right click on each completed sequence present in the qualification, except the Eluent Generator Current Qualification, Flow Rate Accuracy and Precision Qualification, Column Heater Accuracy qualification and the Stop sequence. Select **Batch Report** and **Print** the report.

6. Review the report for Pass/Fail results. If any test does not pass, make the appropriate instrument repair and/or adjustments and rerun all tests from the original point of failure on. Refer to Section 5 for troubleshooting assistance.

3.3.5 Column Heater Temperature Qualification

The column heater temperature qualification can be used to qualify the temperature accuracy of the column heater fitted to an ICS-2000, ICS-1500 or ICS-1000. This qualification does not support AS50 TC, AS3500, LC30 or LC25 column ovens.

1. After completion of all other tests record the reading on the Robo-Thermometer.

2. Open the Water_WU sample in the Warm Up sequence from the browser. Click on Printer Layout from the View menu. Select the Column Heater Temp Qualification tab at the bottom of the page, turn on layout mode and enter the measured temperature in the Measured Plate Temp. cell (cell B43).

   Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

3. The measured liquid temperature and its deviation from theory will be calculated automatically. If the test does not pass, make the appropriate repair/adjustments and rerun the test. Refer to Section 5 for troubleshooting assistance.

4. Print the Column Heater Temperature Verification report by selecting Print… from the File menu. Choose a printer and press OK to begin printing.

3.3.6 Column/Compartment TC Temperature Qualification (ICS-3000 Only)

The column/compartment TC temperature qualification can be used to qualify the temperature accuracy of the column/compartment TC on an ICS-3000. After completion of all other tests record the reading on the Robo-Thermometer.

1. Open the Water_WU sample in the Warm Up sequence from the browser. Click on Printer Layout from the View menu. Select the Temperature Qualification tab at the bottom of the page, turn on layout mode and enter the measured temperature of the Column TC in the "Measured Column Temperature" cell and if the Compartment TC is installed, enter the measured temperature of the Compartment TC in the "Measured Compartment Temperature" cell.

Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

2. If the test does not pass, make the appropriate repair/adjustments and rerun the test. Refer to Section 5 for troubleshooting assistance.

3. Print the Column TC Temperature Verification report by selecting Print… from the File menu. Choose a printer and press OK to begin printing.

3.3.7 EGC reproducibility (ICS-3000 Only – KOH and MSA Only)

The EGC reproducibility test can be used to qualify the concentration reproducibility of a MSA or KOH cartridge when running on an ICS-3000 system. This test should be run as a PQ only on the cartridge and should be run separately from any system tests.

1. Plumb the system with backpressure tubing in place of the column and suppressor (the EG should be plumbed as normal (included degass assymble and CR-TC). The sequence should be run and data evaluated using the OQ/PQ report.

3.3.8 Qualification Comments

The comments section of the report is provided as a place to record any comments, test failures, or configuration changes that occur during the qualification.

To use the comment section, Open the Water_WU sample in the Warm Up sequence from the browser. Click on Printer Layout from the View menu. Select the Comment tab at the bottom of the page, turn on layout mode and enter any comments. This page and also be printed and comments entered manually.

To print the Comment report, select Print… from the File menu. Choose a printer and press OK to begin printing.

Note: A pdf copy of this report is available on the PQ_OQ CD and can be used for manual data collection and data calculations.

3.4 What to do if a Test Fails

If at any point the instrument results do not pass within Dionex specified limits, the instrument must be serviced. See Section 0 for troubleshooting assistance. When the repair is complete, rerun all tests from the original point of failure on in the sequence. All actions for failures and repairs should be recorded on the "comments" report. For example, if the instrument passes tests a, b, and c and fails test d, make the appropriate repairs, record the action, and then repeat tests d – o only.

When rerunning the tests, restart the Instruments OQ or Instrument PQ procedure from the Qualification menu and uncheck all passed tests prior to the test that failed. All tests after the test that failed should be left checked even if they have previously passed. The Warm Up sequence is always required and must be rerun.

3.5 OQ/PQ Completion

3.5.1 Customer Review

1. The printed results should be reviewed by the instrument owner/user.

2. When the qualification is accepted, both the Customer and Qualification Executor need to sign the printed reports.

3. Fill out the Qualification Stickers and Certificates of Functional Performance for the qualified modules.

4. Place the reports and certificates into the binder provided in the OQ/PQ kit and leave it with the customer.

3.5.2 Accuracy of reports

In accordance with Good Laboratory Practice, calculations used to determine the results in the printed reports use more significant figures than those shown and are rounded down for the final report. When reproducing the calculations by hand it may be necessary to obtain numbers from the reports after increasing the number of displayed significant figures.

The number of displayed significant figures can be adjusted through the report publisher. Open the sequence containing the samples for the test being examined and double click on a sample. The sample data should be shown. Under the View menu choose Printer Layout to open the report. Highlight the numbers being used for the hand calculations, click on the Format menu and select number. The number of digits displayed can be increased by changing the number type; for example, entering 0.00000 into the Type field will increase the number of displayed decimal places to 5. **Always use more significant figures for hand calculations than is shown in the printed report.**

3.5.3 Returning the System to the Original Configuration

1. If the Signal factor was modified in the system preparation, or a module was renamed it must be set back to its original value. Open the PeakNet or Chromeleon Server Configuration application. Double click on the detector/s that were qualified to view the properties page. Select the **Signals** page to change the factor back to the original value and the **General** page to change the name back to the original value. Exit the properties page and save the configuration.

2. Disconnect the Backpressure Coil and restore the system plumbing to its original configuration with the appropriate columns, suppressors, and Eluent Generator in line.

3. Remove the sample loop and reinstall the original sample loop back onto the injection valve. For systems using an AS or AS50 Autosampler, go to the **Main Menu** on the front panel of the autosampler. Access the **Module Set Up** screen and then go to **Plumbing Configuration**. Change the sample loop size back to its original value.

4. Reconnect the original eluents to the pump and prime each line individually to purge the eluents used in the OQ/PQ tests.

5. Flush the system with an eluent recommended by the customer and confirm that there are no leaks.

4 Explanation of Test Procedures

4.1 Flow Rate Accuracy and Precision Qualification

4.1.1 Theory

The accuracy and precision of a pump is qualified by accurately measuring 5 aliquots of eluent into a tared sample beaker for exactly 5 minutes each. The injection valve is used to measure the 5 minute aliquot and the volume of each aliquot is determined gravimetrically. The backpressure coil is placed before the injection valve and equal lengths of tubing are used for eluent and waste to ensure that there is no backpressure change when switching the valve from waste to sample.

4.1.2 Performing the Test and Evaluating the Results

The Flow Rate Accuracy and Precision Qualification is the final test in the sequence batch and the sequence is called XQ_Flow_Rate. The five weight measurements for the aliquots are entered manually into the report and converted to volume when the temperature is entered. Accuracy is measured by comparing the theoretical value (5.00 mL in 5.00 min) to each of the five aliquots and precision is measured by determining the relative standard deviation of the five aliquots.

4.2 Eluent Generator Current Qualification

4.2.1 Theory

Eluent Generator Current Qualification is determined by performing an EG step gradient using varying eluent concentration set points. The Eluent Generator applies a specific current to the EluGen cartridge depending on the concentration of eluent and type of cartridge selected. An IC validation/test box is used in place of the EluGen cartridge and the applied current for each step is measured using an ammeter or voltmeter that is connected to the test cell. No injection is performed.

4.2.2 Performing the Test and Evaluating the Results

The Eluent Generator current qualification test is the next test in the sequence batch and the sequence is called XQ_EG_Current or XQ_EG_Dual_Current. This is Test k in the OQ/PQ test order. Current readings in mA are displayed on the voltmeter for each concentration step in the gradient. Values for each step are manually recorded; therefore, the sequence follows an XQ_Stop sequence that will pause the system to allow connection of the validation cell and recording of the results.

4.3 Wavelength Accuracy of Absorbance Detectors

4.3.1 Theory

Wavelength accuracy is evaluated using a holmium oxide filter. Three holmium oxide maxima are compared with the theoretical maxima at 360.9 nm, 418.7 nm and 536.2 nm for AD25 and PDA-100 detectors. Absorbance readings are measured with a holmium oxide filter at varying wavelength settings for the AD20 wavelength accuracy test. The wavelength accuracy test is not conducted for UVD-170U and UVD-340U detectors.

4.3.2 Performing the Test and Evaluating the Results

For AD25 and PDA-100 detectors the wavelength accuracy test is the first test in the sequence batch and is called XQ_Wavelength_Accuracy where, X is P for PQ and O for OQ. This is test c in the OQ/PQ test order. The Wavelength Accuracy Test program executes the verification and the results are logged to the audit trail by the Wavelength Accuracy Report program.

The test is performed from the front panel of the AD20 by setting wavelengths manually and recording the resulting absorbance values. This is test a in the OQ/PQ test order.

4.4 Warm up

The warm up sequence is used to ensure that all parts of the system are responding appropriately and to record logs to the audit trail (later used to update the information in the report definition file). It is called XQ_Warm_Up where; X is P for PQ and O for OQ. (For DX-320 and DX-120 systems, an IC prefix will precede Warm_Up). The warm up sequence is compulsory for all qualifications except the amperometry detector qualifications where no warm up is required. This is Test b in the OQ/PQ test order.

4.5 Baseline Noise and Drift

4.5.1 Theory

A 20-minute detector baseline is collected using water as the eluent at a flow rate of 1.00 mL/min. For AD20, AD25 and PDA-100 detectors, data is collected at 254 nm and 520 nm; for UVD-170U and UVD-340U, data is collected at 254 nm only. No injection is performed.

4.5.2 Performing the Test and Evaluating the Results

Noise and drift evaluation for all IC detectors is the second test in the sequence batch (first for conductivity detectors) and is called XQ_Detector_Noise_Drift where, X is P for PQ and O for OQ and Detector is CD for conductivity detectors, UV_Vis for AD20/25 detectors and PDA for PDA-100, UVD-170U and UVD-340U detectors (For DX-320 and DX-120 systems, an IC prefix will precede the Detector specification). This is Test d in the OQ/PQ test order.

Noise is calculated by splitting the 20-minute baseline signal into 20 one-minute intervals. The noise is the distance between two parallel lines through the measured minimum and maximum values and the regression line. The measured noise values are then averaged to establish the final value. To calculate drift, the Dionex Chromatography Management System uses a method of least squares and calculates a regression line from the measured values. The slope of the curve indicates the drift of the measured signal, and the amount of the slope indicates the amount of drift.

4.6 DC Amperometry Noise and Drift (DX500/DX600/ICS-2500)

4.6.1 Theory

A 20-minute detector baseline is collected using an amperometry validation test cell. No injection is carried out and the flow rate is set to 0.00 mL/min.

4.6.2 Performing the Test and Evaluating the Results

Noise and drift evaluation for DC Amperometry detectors is the only test in the sequence batch and is called XQ_DC_Amp_Noise_Drift. This is Test d in the OQ/PQ test order.

Noise is calculated by splitting the 20-minute baseline signal into 20 one-minute intervals. The noise is the distance between two parallel lines through the measured minimum and maximum values and the regression line. The measured noise values are then averaged to establish the final value. To calculate drift, the Dionex Chromatography Management System uses a method of least squares and calculates a regression line from the measured values. The slope of the curve indicates the drift of the measured signal, and the amount of the slope indicates the amount of drift.

4.7 Integrated Amperometry Noise and Drift (DX500/DX600/ICS-2500)

4.7.1 Theory

A 20-minute detector baseline is collected using an amperometry validation test cell. No injection is carried out and the flow rate is set to 0.00 mL/min.

4.7.2 Performing the Test and Evaluating the Results

Noise and drift evaluation for Integrated Amperometry detectors is the only test in the sequence batch and is called XQ_Int_Amp_Noise_Drift. This is Test d in the OQ/PQ test order.

Noise is calculated by splitting the 20-minute baseline signal into 20 one-minute intervals. The noise is the distance between two parallel lines through the measured minimum and maximum values and the regression line. The measured noise values are then averaged to establish the final value. To calculate drift, the Dionex Chromatography Management System uses a method of least squares and calculates a regression line from the measured values. The slope of the curve indicates the drift of the measured signal, and the amount of the slope indicates the amount of drift.

4.8 Integrated Amperometry Noise and Drift (ICS-3000)

4.8.1 Theory

A 20-minute detector baseline is collected using 50 mM NaOH as the eluent at a flow rate of 0.25 mL/min. An Amino Acid disposable gold working electrode (Dionex Part Number 060082) is used running the Amino Acids (pH,Ag,AgCl Reference) waveform. No Injection is performed.

4.8.2 Performing the Test and Evaluating the Results

Noise and drift evaluation for Integrated Amperometry detectors is the only test in the sequence batch and is called XQ_ED_Amp_Noise_Drift. This is Test d in the OQ/PQ test order.

Noise is calculated by splitting the 20-minute baseline signal into 20 one-minute intervals. The noise is the distance between two parallel lines through the measured minimum and maximum values and the

regression line. The measured noise values are then averaged to establish the final value. To calculate drift, the Dionex Chromatography Management System uses a method of least squares and calculates a regression line from the measured values. The slope of the curve indicates the drift of the measured signal, and the amount of the slope indicates the amount of drift.

4.9 Injector Precision

4.9.1 Theory

Injector precision is determined by making ten replicate injections of a standard using a fixed sample volume. A 40 ppm caffeine standard is injected when using absorbance detection at a wavelength of 272 nm. A 50 ppm nitrate standard is injected when using conductivity detection. Deionized water is used as the eluent at a flow rate of 1.00 mL/min. A 10 uM threonine standard is injected when using ICS-3000 ED detector in Integrated Amperometry mode. 50 mM NaOH is used as eluent at a flow rate of 0.25 mL/min. An Amino Acid disposable gold working electrode (Dionex Part Number 060082) is used running the Amino Acids (pH,Ag,AgCl Reference) waveform.

4.9.2 Performing the Test and Evaluating the Results

Injector precision evaluation is the next test in the sequence batch and the samples are called Inj Precision_1 through Inj Precision_10 in the XQ_Detector_Inj_Precision sequence, where X is O for OQ, P for PQ, and Detector is CD for conductivity detectors, Amp for amperometry detectors, ED is for ICS-3000 systems with amperometry detector . (For DX-320 and DX-120 systems, an IC prefix will precede the Detector specification). This is Test e in the OQ/PQ test order. The concentration of the nitrate or caffeine standard and the sample volume injected is included in the QNT file used for injector linearity.

Precision is determined by calculating the relative standard deviation of the peak areas for the ten injections.

4.10 Sample Carry-over

4.10.1 Theory

Sample carry-over is determined by injecting a highly concentrated sample, followed by a blank sample then a second blank injection. The blanks in this case are water. Nitrate at a concentration of 1000 ppm is injected when this test is performed with a conductivity detector. Caffeine at a concentration of 250 ppm is injected when this test is performed with an absorbance detector and a 25 µL loop. Caffeine at a concentration of 80 ppm is injected when this test is performed with an absorbance detector and a 100 µL loop. Threonine at a concentration of 10uM is injected when this test is performed with an ICS-3000 with ED detector in Integrated Amperometry mode. Ideally, the chromatogram of the two blank injections should be identical. If, however, a difference is detected, the difference between the peak areas is calculated and the ratio of this difference to the highly concentrated sample is calculated. Deionized water is used as the eluent at a flow rate of 1.00 mL/min.

4.10.2 Performing the Test and Evaluating the Results

Sample carry-over evaluation is included in the same sequence as the Injector Precision test of the sequence batch and the samples are called Water, Carry Over and Water_CO in the XQ_Detector_Inj_Precision_CO sequence. This is Test f in the OQ/PQ test order.

A peak area ratio is calculated to determine sample carry-over.

4.11 Detector Linearity

4.11.1 Theory

Detector linearity for absorbance detectors is determined at 272 nm using a sequence of five caffeine standard injections at 15, 40, 60, 80 and 100 ppm. Linearity for conductivity detectors uses a sequence of five nitrate standard injections at 5, 10, 25, 50, and 100 ppm. Deionized water is used as the eluent at a flow rate of 1.00 mL/min. . Linearity for ICS-3000 Integrated Amperometry detectors uses a sequence of five threonine standard injections at 1, 2, 4, 8, and 10 uM. 50 mM NaOH is used as the eluent at a flow rate of 0.25 mL/min.

4.11.2 Performing the Test and Evaluating the Results

Detector linearity evaluation is the next test in the sequence batch and the samples are called Detector linearity_1 through Detector linearity_5 in the XQ_Detector_Linearity sequence. This is Test g in the OQ/PQ test order. The concentrations for the nitrate and caffeine standards are included in the QNT file used for detector linearity.

4.12 Injection Volume Linearity

4.12.1 Theory

Injection volume linearity is determined by making five injections of a standard using injection volumes of 10, 20, 30, 40 and 50 µL. A 60 ppm caffeine standard is injected when using absorbance detection at a wavelength of 272 nm. A 50 ppm nitrate standard is injected when using a conductivity detection. Deionized water is used as the eluent at a flow rate of 1.00 mL/min. A 8 uM threonine standard is injected when using integrated amperometry detection. 50 mM NaOH is used as the eluent at a flow rate of 0.25 mL/min This test is only performed on autosamplers with variable injection volume capability (AS, AS50 and TSP AS3500).

4.12.2 Performing the Test and Evaluating the Results

Injection volume linearity evaluation is the next test in the sequence batch and the sequence is called XQ_Detector_Inj_Linearity. This is Test h in the OQ/PQ test order. The concentrations for the nitrate and caffeine standard and the injection volumes are included in the QNT file used for detector linearity.

4.13 Sample Preparation Precision

4.13.1 Theory

Sample Prep precision is determined by making three separate injections using a fixed injection volume. Each of the three standards is prepared individually using the autosampler's sample preparation option. The autosampler prepares the three samples by first transferring 15 µL of a 1000 ppm caffeine or nitrate standard into three empty sample vials and then adding 1485 µL of water to each. Injections are then made from each vial. Deionized water is used as the eluent at a flow rate of 1.00 mL/min. If an absorbance detector is used the wavelength is set to 272 nm. This test is only performed on autosamplers with the sample prep option installed.

4.13.2 Performing the Test and Evaluating the Results

Sample prep precision evaluation is the next test in the sequence batch and the sequence is called XQ_Detector_Samp_Prep. This is Test i in the OQ/PQ test order.

Precision is determined by calculating the relative standard deviation of the peak areas for the three injections.

4.14 Gradient Accuracy

4.14.1 Theory

Gradient accuracy is determined by performing a step gradient using varying compositions of deionized water and either 0.3% Acetone or 1 mM KCL. When performing the test with an absorbance detector, deionized water is used for eluents A and C and 0.3% Acetone is used for eluents B and D with a wavelength setting of 254 nm. When performing the test with a conductivity detector, deionized water is used for eluents A and C and 1 mM KCl is used for eluents B and D. Gradient composition is measured at 0, 20, 50, 80, and 100% of eluents B and D at a flow rate of 1.00 mL/min (0.75 mL/min for microbore pumps). Step noise (ripple) is measured for each step in the gradient. No injection is performed.

4.14.2 Performing the Test and Evaluating the Results

Gradient Accuracy evaluation is the next test in the main part of the sequence batch and the sequence is called XQ_GP_Grad_Accuracy. This is Test j in the OQ/PQ test order.

The measured step height is compared to the theoretical step height and a percent deviation is calculated. Step noise is evaluated for all steps. Each step has a defined one-minute interval where noise is evaluated. Based on a method of least squares, PeakNet™ or Chromeleon calculates a regression line from the measured values of each interval. The noise is the distance between two parallel lines through the measured minimum and maximum values and the regression line.

4.15 Column Heater Temperature Qualification

4.15.1 Theory

The temperature of a column heater is qualified by measuring the plate temperature of the column heater after allowing the column heater plate and thermometer to equilibrate for a minimum of 15 minutes. The temperature is read off the thermometer, giving the temperature of the column heater plate. The temperature displayed by the front panel and the software is the liquid temperature, which can be calculated from the plate temperature.

4.15.2 Performing the Test and Evaluating the Results

During the sequence batch the column heater is commanded to 40°C. The Column Heater Temperature Qualification is performed after the completion of the sequence batch. The recorded plate temperature is entered into the report and automatically converted to liquid temperature. The calculated liquid temperature is compared to the theoretical value (40°C) and a pass or fail criteria applied.

4.16 Column/Compartment TC Temperature Qualification

4.16.1 Theory

The temperature of a column/compartment TC is qualified by measuring the temperature of the column/compartment TC after allowing the column/compartment TC thermometer to equilibrate for a minimum of 15 minutes. The temperature is read off the thermometer, giving the temperature of the column/compartment TC. The temperature displayed by the front panel and the software is the actual liquid temperature.

4.16.2 Performing the Test and Evaluating the Results

During the sequence batch the column heater is commanded to 30°C. The Column Heater Temperature Qualification is performed after the completion of the sequence batch. The recorded temperature is entered into the report.

4.17 Eluent Generator Cartridge Reproducibility Qualification

4.17.1 Theory

Eluent Generator Cartrige Reproducibility Performance Qualification is determined by performing an EG step gradient using varying eluent concentration set points. The Eluent Generator applies a specific current to the EluGen cartridge depending on the concentration of eluent and type of cartridge selected. The concentration is monitored by changes in conductivity. The EG step gradient is run three times and the reproducibility is evaluated. No injection is performed.

4.17.2 Performing the Test and Evaluating the Results

The Eluent Generator current qualification test is the next test in the sequence batch and the sequence is called PQ_EG_Cartridge Reproducibility. This is Test q in the PQ test order. Current readings in uS are displayed on the conductivity detector for each concentration step in the gradient. Values for each step are automatically recorded in Chromeleon.

4.18 General Hints

If the system pressure is above 2000 psi after connecting the PEEK Backpressure coil with a flow rate of 1.00 mL/min, the coil has a blockage. Recut the ends of the coil or replace it.

Dips before or after the main nitrate or caffeine peak indicate extra dead volume in the system. This is caused by small voids between the end of a liquid line and the bottom of the port. Individually disconnect each line from the injection valve to the detector cell. Push the ferrule and fitting up and away from the end of the tubing. Make sure the tubing is cut straight. If it is not, recut the end. When reinstalling all liquid connections, first insert the tubing all the way into the port until it touches the bottom. Hold the tubing in this position while inserting and tightening ferrule and fitting down into the port.

4.19 Failure of Individual Tests

4.19.1 Absorbance Detector

Test	Reason	Action
Wavelength Accuracy	Optics not calibrated	Perform wavelength calibration and then repeat the wavelength accuracy test
	Air bubbles in flow cell	Flush out flow cell
	Colored reagent in flow cell	Flush out flow cell
Baseline Noise	Poor water quality	Replace with an alternate water source
	Lamp is reaching maximum lifetime	Replace lamp
	Air bubbles in flow cell	Flush out flow cell
Drift	Detector not warmed up	Allow recommended warm up period (refer to test procedure)
	System not equilibrated	Allow longer flush/equilibration time
	Lamp defective	Replace lamp
	Water not degassed	Degas water or turn degas unit onto "Always On" setting.
Lamp Intensity (PDA-100 only)	Lamp is reaching maximum lifetime	Replace lamp
	Flow cell inserted incorrectly	Reinstall flow cell
Detector Linearity	Lamp is reaching maximum lifetime	Replace lamp
	Concentration of standards is incorrect	Obtain new fresh standards
	Liquid leak	Find and repair source of leak

4.19.2 Conductivity Detector Tests

Test	Reason	Action
Baseline Noise	Poor water quality	Replace with an alternate water source
	Air bubbles in flow cell	Flush out flow cell
	Cell temperature regulation failure	Replace cell
Drift	System not equilibrated	Allow longer flush/equilibration time
	Lab temperature not stable	Regulate temperature (block vents, close windows, etc.)
	Water not degassed	Degas water or turn degas unit onto "Always On" setting.
Detector Linearity	Concentration of standards is incorrect	Obtain new fresh standards
	Liquid leak	Find and repair source of leak
	Injection volume not consistent (manual injections only)	Overfill sample loop at least 3x when loading

4.19.3 Pump

Test	Reason	Action
Gradient Step Accuracy	Air in system	Prime pump – prime each eluent line individually for 3 – 5 minutes, flush system
	System not equilibrated	Allow longer flush/equilibrium time
	Old eluent in vacuum degas chamber	Prime pump – prime each line individually for 3 – 5 minutes
	Incorrect eluent concentration for eluent B and D	Remake 0.3% Acetone or 1 mM KCl solutions
	GP40 pump eluents not pressurized	Pressurize eluent bottles with 5 psi helium
	GP50/GS50 eluent bottles not vented	Vent eluent bottles
Step Noise (ripple)	Air in system	Prime pump, flush system
	Air in flow cell	Flush flow cell with iso-propyl alcohol, ensure adequate backpressure on flow cell
	System not equilibrated	Allow longer flush/equilibrium time
	Check valve or piston seal failure	Replace check valve/piston seal
	Void time from pump to detector too long or short	Adjust tubing length accordingly

Flow Rate Accuracy and Precision	Flow rate out of calibration	Re-calibrate the flow rate of the pump

4.19.4 Autosampler

Test	Reason	Action
Injector Precision	Air injected from vial or from sample syringe when doing manual injections	Too little sample volume in vial – refill vial or flush bubbles out of injection syringe
	Air bubbles in AS sample syringe	Flush out syringe
	Liquid leak in autosampler or injection valve	Find and repair source of leak
	Poor water quality in flush reservoir	Change water and flush sampler fluidics
Injection Volume Linearity	Detector failure	See Detector linearity troubleshooting section
	Defective sample syringe	Replace sample syringe
Carry Over	Contaminated injection valve	Replace injection valve rotor and stator face
	Contamination in autosampler	Flush/replace autosampler liquid lines
	Contamination in sample vial or in syringe used for manual injections	Use new sample vial for deionized water blank, rinse vial thoroughly before using, flush syringe or use a new one
Sample Prep Precision	Air injected from vial	Too little sample volume in vial, refill vial
	Air bubbles in AS sample syringe or prep syringe	Flush out syringe
	Defective prep syringe	Replace prep syringe
	Liquid leak in autosampler or injection valve	Find and repair source of leak
	Poor water quality in Sample Prep reservoir	Change water and flush sampler fluidics

4.19.5 DC and Integrated Amperometry Detector Tests

Test	Reason	Action
Baseline Noise	Validation cell not connected properly	Reconnect test cell cable to amperometry cell connection on SP board
	Validation cell defective	Replace validation test cell
Drift	Detector not equilibrated	Allow longer equlibration time
	Lab temperature not stable	Regulate temperature (block vents, close windows, etc.)

4.19.6 Eluent Generator (EG40, EG50, ICS-2000 and ICS-3000 EG)

Test	Reason	Action
Current	Validation/Test box not connected properly	Reconnect test cell cable to the Eluent Generator, confirm ammeter or multimeter leads are making contact with test cell wires
	Ammeter or multimeter on wrong setting	Ammeter or multimeter should be set to read current (mA). Range should be set to view 0.00 to 300.00 mA
	Validation cell defective	Replace validation test cell

4.19.7 Column Heater (ICS-3000, ICS-2000, ICS-1500 and ICS-1000, if fitted)

Test	Reason	Action
Temperature Qualification	Column Heater not calibrated	Recalibrate the Column Heater
	Robo-Thermometer not calibrated	Obtain a new calibrated Robo-Thermometer
	Thermometer not fitted in column heater (compartment) correctly	Fit thermometer in column heater (compartment) correctly

5 Example Reports

Following are two example PQ Reports. The first report is for an ICS-3000 system configured with an CD Detector, DP Gradient Pump, and an AS AutoSampler. The second report is for an ICS-2000 system with a MSA EluGen cartridge and an AS50 AutoSampler.

The OQ and PQ reports are the same except the test limits differ as shown on page 10.

DIONEX

Operational Qualification

Instruments

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradient)	Dionex	5090230	2.72
Thermal or Detector Compartment	ICS-3000 DC	Dionex	05100169	1. 3. 1
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1
Fluorescence Detector	n.a.	Dionex	n.a.	n.a.
Autosampler	AS	Dionex	05090727	2.01
Eluent Generator	ICS-3000 EG (EluGen-OH)	Dionex	05100934	1. 3. 0
Chromatography Datasystem	6.80 SP1 Build 2232 (Beta) (Demo-Installation)	Dionex	29461	n.a.

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*	*Exp. Date*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")			
Blank	Water			
ECD Sample 1	Nitrate, 5 ppm			
ECD Sample 2	Nitrate, 10 ppm			
ECD Sample 3	Nitrate, 25 ppm			
ECD Sample 4	Nitrate, 50 ppm			
ECD Sample 5	Nitrate, 100 ppm			
ECD Sample 6	Nitrate, 1000 ppm			
Eluent A and C	Water			
Eluent B and D	1.0 mM KCl			
Autosampler Reservoir A	Water			
Balance				
Temperature Probe				
EG Validation Test Cell				
Ammeter / Multimeter				

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Customer signature indicates that all information in the following reports has been reviewed and accepted.

Limits

Test	Customized Limits	Dionex Recommended Limits
Conductivity Noise (nS)	<= 1.0	<= 1.0
Conductivity Drift (nS/hr)	<= 10	<= 10
Injector Precision (Area %RSD)	<= 0.8	<= 0.8
Injector Carry Over (Area %)	<= 0.10	<= 0.10
Conductivity Linearity (Corr.)	>= 0.999	>= 0.999
Conductivity Linearity (%RSD)	<= 5.0	<= 5.0
Injector Linearity (Corr.)	>= 0.999	>= 0.999
Injector Linearity (%RSD)	<= 1.0	<= 1.0
Eluent Generator Step Current (mA)	See Worksheet(s)	See Worksheet(s)
Pump Gradient Accuracy (%)	<= 2.0	<= 2.0
Pump Noise (%)	<= 0.2%	<= 0.2%
Pump Flow Accuracy (mL/min)	<= 0.05	<= 0.05
Pump Flow Precision (%RSD)	<= 2.0%	<= 2.0%
Heater Temperature Verification	<= 2.0	<= 2.0

Additional Information

Customer/Company: **Mr. Customer/ Dionex**

Date: **18-May-2006**

Qualification Executor/Company: **Mr. Citizen / Dionex**

Period between Qualifications: **6 months**

Next Qualification: **Nov-06**

Customer Signature _____ Date _____

Qualification Executor _____ Date _____

DIONEX

Operational Qualification

Conductivity Detector Noise and Drift:

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradi	Dionex	5090230	2.72
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
Eluent	Water		

• *Additional Information*

Customer/Company:	**Mr. Customer/ Dionex**	Date:	**05-17-06**
Qualification Executor/Company:	**Mr. Citizen / Dionex**	Next Qualification Due:	**Nov-06**

• *Test Results Summary*

Test	*Result*
Noise	***PASS***
Drift	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• Data for Detector Noise

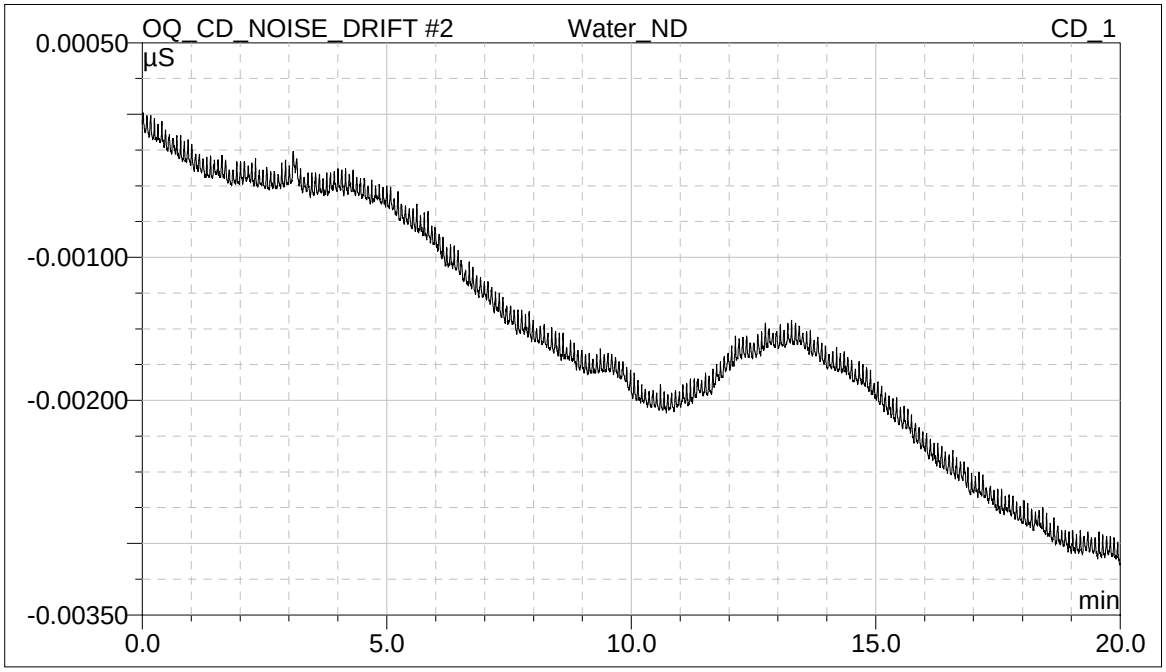
Segment No.	Noise, nS
1	0.1830355566
2	0.197184476
3	0.225776796
4	0.2827726199
5	0.2049540725
6	0.2200292093
7	0.2056566296
8	0.232684102
9	0.2063104629
10	0.2381187009
11	0.2242075941
12	0.2190239313
13	0.2252697098
14	0.238267809
15	0.1993815749
16	0.1978368848
17	0.2126491508
18	0.1965705065
19	0.2035378476
20	0.2026084559
Average, nS	**0.216**
Limit, nS	**1**
Result	***PASS***

• Data for Detector Drift

20 Minute Drift, nS	Drift, nS/hr	Limit, nS/hr	Result
-2.842	8.527	10	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• *Chromatogram of Detector Noise and Drift*



Customer Signature	Date	Qualification Executor	Date

DIONEX

Operational Qualification

Injector Precision

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradient)	Dionex	5090230	2.72
Thermal or Detector Compartment	ICS-3000 DC	Dionex	05100169	1. 3. 1
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1
Autosampler	AS	Dionex	05090727	2.01

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
ECD Sample 4	Nitrate, 50 ppm		
Eluent A and C	Water		

• *Additional Information*

Customer/Company:	**Mr. Customer/ Dionex**	Date:	**17-May-2006**
Qualification Executor/Company:	**Mr. Citizen / Dionex**	Next Qualification:	**Nov-06**

• *Test Results Summary*

Test	*Result*
Injector Precision (Area %RSD)	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• *Data for Injector Precision Test* *25.0 µl*

	Injector Precision
Name	***Area µS*min CD_1***
Inj Precision_1	2.756
Inj Precision_2	2.769
Inj Precision_3	2.807
Inj Precision_4	2.803
Inj Precision_5	2.800
Inj Precision_6	2.790
Inj Precision_7	2.818
Inj Precision_8	2.813
Inj Precision_9	2.808
Inj Precision_10	2.797
Average:	***2.796***
Std Dev:	***0.020***
% RSD:	***0.701 %***
Limit:	***0.8 %***
Result:	***PASS***

Customer Signature Date

Qualification Executor Date

Operational Qualification

Injector Carry Over

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradien	Dionex	5090230	2.72
Thermal or Detector Compartment	ICS-3000 DC	Dionex	05100169	1. 3. 1
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1
Autosampler	AS	Dionex	05090727	2.01

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
ECD Sample 6	Nitrate, 1000 ppm		
Blank	Water		
Eluent A and C	Water		

• *Additional Information*

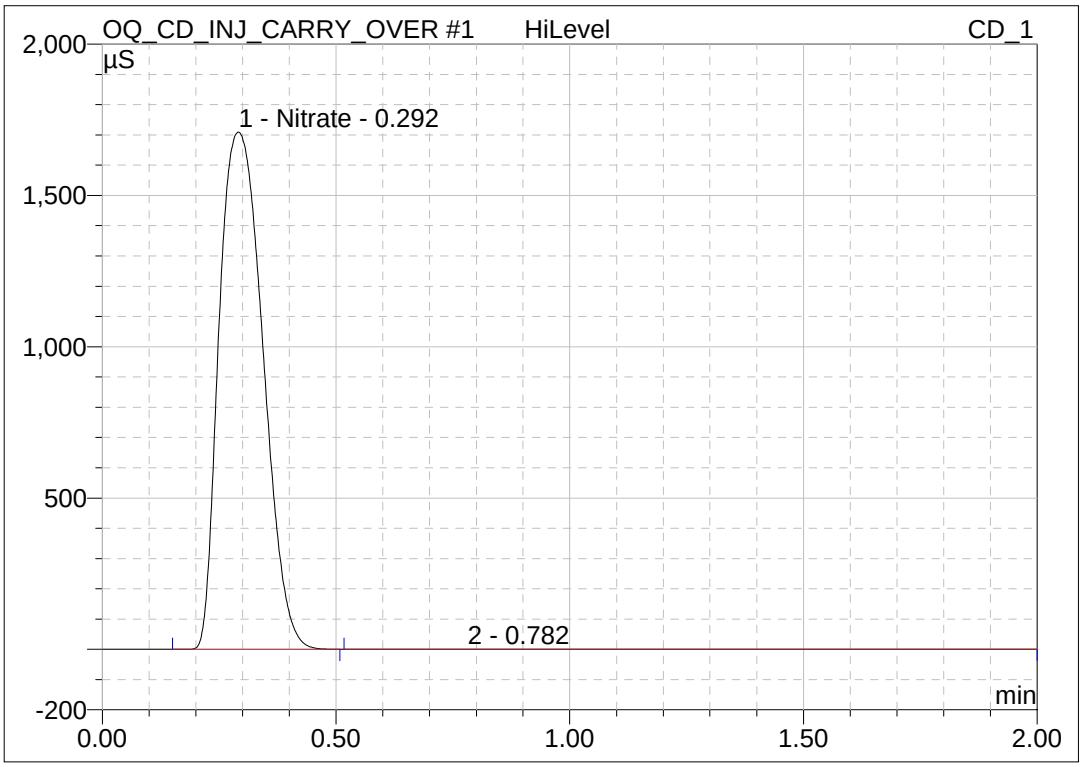
Customer/Company:	**Mr. Customer/ Dionex**	Date:	**18-May-2006**
Qualification Executor/Company:	**Mr. Citizen / Dionex**	Next Qualification:	**Nov-06**

• *Test Results Summary*

Test	*Result*
Injector Carry Over (Area %)	***PASS***

Customer Signature Date Qualification Executor Date

• *Chromatogram for Carry Over Test*



• *Data for Carry Over Test*

Name	***Ret.Time (detected)*** ***min*** ***CD_1***	***Area*** ***µS*min*** ***CD_1***
HiLevel	0.292	185.639
Carry Over	0.295	0.632
Water	0.295	0.607
Carry over:		***0.014 %***
Limit:		***0.1 %***
Result:		***PASS***

DIONEX

Operational Qualification

Detector Linearity

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradient)	Dionex	5090230	2.72
Thermal or Detector Compartment	ICS-3000 DC	Dionex	05100169	1. 3. 1
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1
Autosampler	AS	Dionex	05090727	2.01

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
ECD Sample 1	Nitrate, 5 ppm		
ECD Sample 2	Nitrate, 10 ppm		
ECD Sample 3	Nitrate, 25 ppm		
ECD Sample 4	Nitrate, 50 ppm		
ECD Sample 5	Nitrate, 100 ppm		
Eluent A and C	Water		

• *Additional Information*

Customer/Company: **Mr. Customer/ Dionex**

Date: **05-18-06**

Qualification Executor/Company: **Mr. Citizen / Dionex**

Next Qualification: **Nov-06**

• *Test Results Summary*

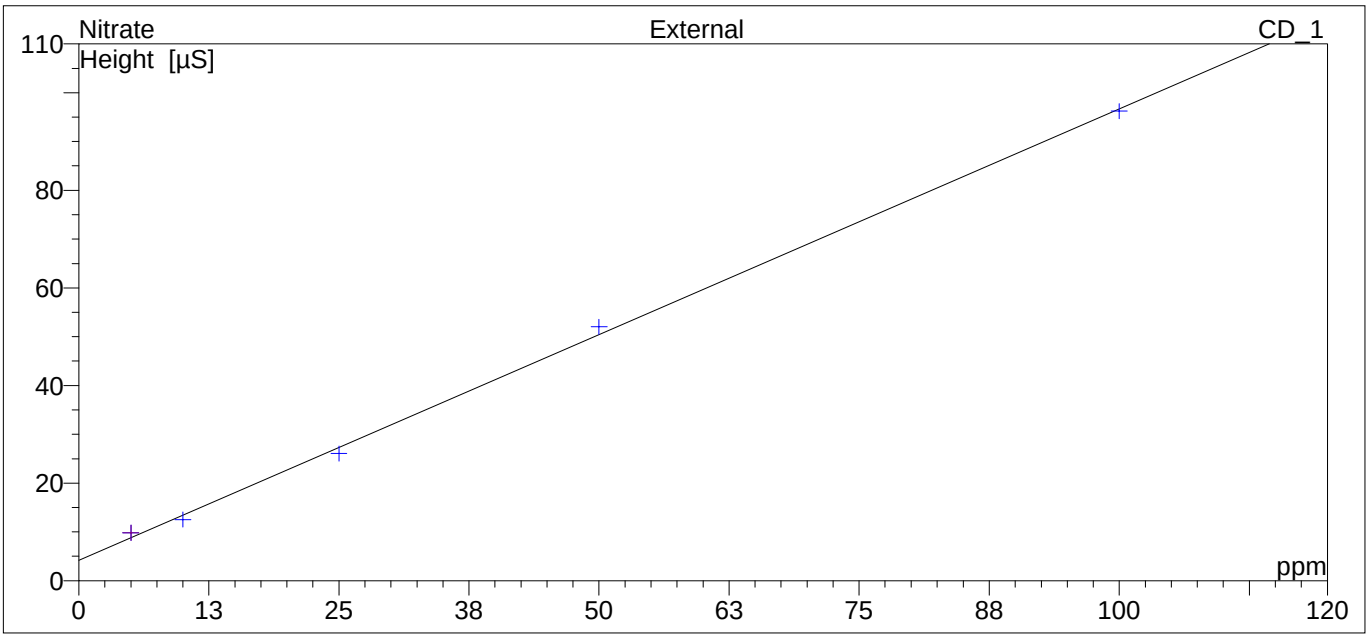
Test	*Result*
Conductivity Linearity (Corr.)	***PASS***
Conductivity Linearity (%RSD)	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• Data for Detector Linearity

Sample Name	Amount ppm Nitrate CD_1	Height µS CD_1	Residual CD_1
Detector linearity_1	5.0000	9.8292	1.0044
Detector linearity_2	10.0000	12.5417	-0.9060
Detector linearity_3	25.0000	26.0686	-1.2480
Detector linearity_4	50.0000	52.0258	1.5944
Detector linearity_5	100.0000	96.2160	-0.4448
		Variance	2.042
		Standard Deviation	1.429

• Linearity Plot



Cal.Type	Number of Points	Offset	Slope
LOff	5	4.202	0.925

	Correlation Coefficient	% RSD
Linearity	0.9994	3.633 %
Limit:	0.999	5 %
Result:	PASS	PASS

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Operational Qualification

Injector Linearity

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Gradient)	Dionex	5090230	2.72
Thermal or Detector Compartment	ICS-3000 DC	Dionex	05100169	1. 3. 1
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1
Autosampler	AS	Dionex	05090727	2.01

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
ECD Sample 4	Nitrate, 50 ppm		
Eluent A and C	Water		

• *Additional Information*

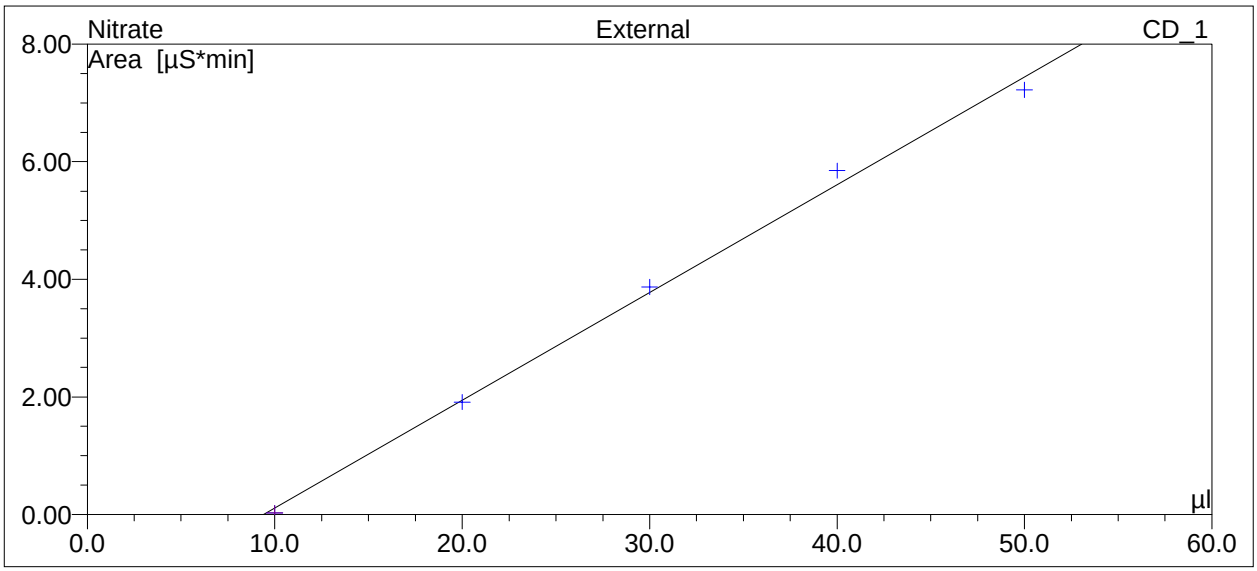
Customer/Company:	**Mr. Customer/ Dionex**	Date:	**05-18-06**
Qualification Executor/Company:	**Mr. Citizen / Dionex**	Next Qualification:	**Nov-06**

• *Test Results Summary*

Test	*Result*
Injector Linearity (Corr.)	***FAIL***
Injector Linearity (%RSD)	***FAIL***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• *Linearity Plot*



• *Data for Injector Linearity Test*

Name	*Ret.Time (detected)* *min* *CD_1*	*Inj.Vol.* *µL*	*Area* *µS*min* *CD_1*	*Residuals* *CD_1*
Injector Linearity_1	0.27	10.0	0.026	-0.0847
Injector Linearity_2	0.27	20.0	1.913	-0.0300
Injector Linearity_3	0.28	30.0	3.871	0.0949
Injector Linearity_4	0.29	40.0	5.847	0.2390
Injector Linearity_5	0.29	50.0	7.221	-0.2192
			Variance	0.0408
			Standard Deviation	0.2019

Cal.Type	*Offset*	*Slope*	*#Points*
LOff	-1.722	0.183	5

	Correlation Coefficient	*% RSD*
Injector Linearity	0.9982	5.347
Limit:	***0.999***	***1.0 %***
Result:	***FAIL***	***FAIL***

Customer Signature _____ Date _____

Qualification Executor _____ Date _____

DIONEX

Operational Qualification

Gradient Pump Accuracy and Noise

• *Instruments and Fluidics*

Instrument name	*Model*	*Supplier's Name*	*Serial Number*	*Moduleware Version*
Pump	ICS-3000 DP (Grad	Dionex	5090230	2.72
Absorbance Detector	n.a.	Dionex		
Electrochemical Detector	ICS-3000 CD	Dionex	05101111	1. 3. 1

• *Accessories*

Name	*Description*	*Vendor*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
Eluent A and C	Water		
Eluent B and D	1 mM KCL		

• *Additional Information*

Customer/Company: **Mr. Customer/ Dionex** Date: **05-18-06**

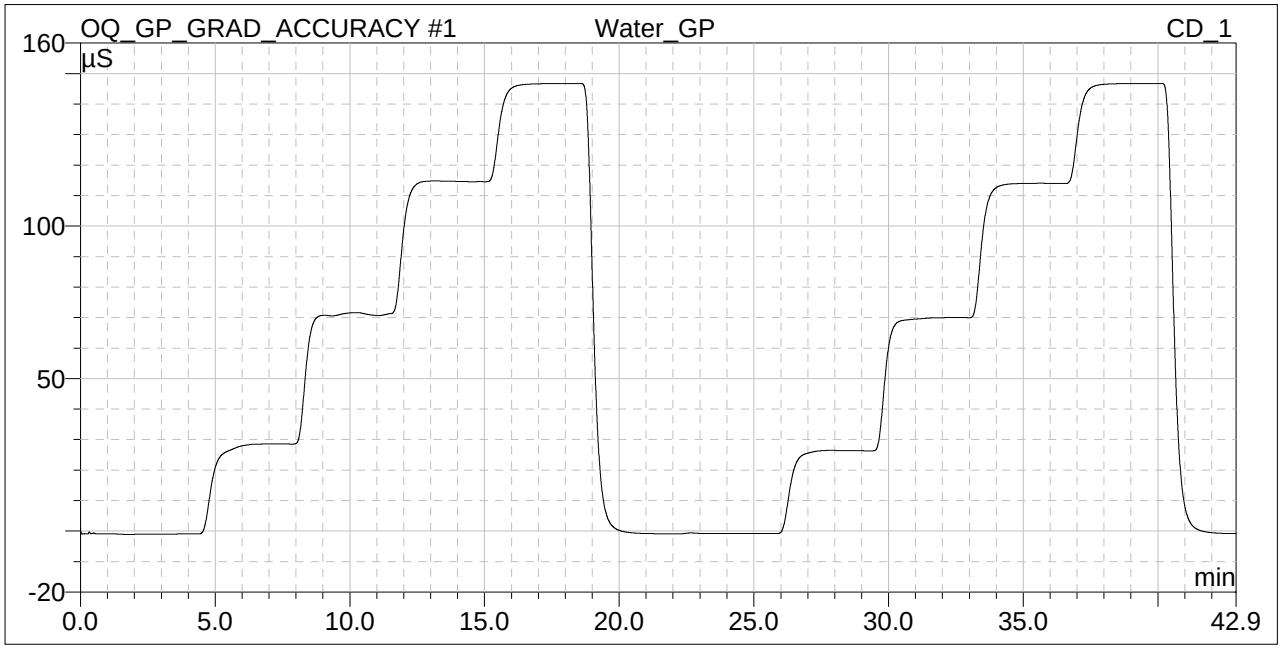
Qualification Executor/Company: **Mr. Citizen / Dionex** Next Qualification: **Nov-06**

• *Test Results Summary*

Test	*Result*
Step Accuracy - A, B	***PASS***
Step Accuracy - C, D	***FAIL***
Step Noise - A, B	***FAIL***
Step Noise - C, D	***PASS***

Customer Signature Date Qualification Executor Date

• *Chromatogram of Gradient Pump Accuracy*



• *Step Accuracy - Valves A, B*

Theoretical Value (%)	*Conductivity (µS)*	*Calculated Value (%)*	*Deviation (%)*	*Limit (+/- , %)*	*Result*
0.00	-1.00	0.00	0.00	N/A	*----*
20.00	28.51	19.98	-0.02	2.0	*PASS*
50.00	70.98	48.74	-1.26	2.0	*PASS*
80.00	114.55	78.24	-1.76	2.0	*PASS*
100.00	146.69	100.00	0.00	2.0	*PASS*

• *Step Accuracy - Valves C, D*

Theoretical Value (%)	*Conductivity (µS)*	*Calculated Value (%)*	*Deviation (%)*	*Limit (+/- , %)*	*Result*
0.00	-0.86	0.00	0.00	N/A	*----*
20.00	26.36	18.45	-1.55	2.0	*PASS*
50.00	69.94	47.98	-2.02	2.0	*FAIL*
80.00	113.99	77.83	-2.17	2.0	*FAIL*
100.00	146.71	100.00	0.00	2.0	*PASS*

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

• Step Noise - Valves A, B

Step (%)	Noise (µS)	Calculated Noise (%FS)	Limit (%)	Result
0.00	0.00	0.00%	0.20%	**PASS**
20.00	0.05	0.04%	0.20%	**PASS**
50.00	0.60	0.41%	0.20%	**FAIL**
80.00	0.08	0.05%	0.20%	**PASS**
100.00	0.02	0.01%	0.20%	**PASS**

• Step Noise - Valves C, D

Step (%)	Noise (µS)	Calculated Noise (%FS)	Limit (%)	Result
0.00	0.00	0.00%	0.20%	**PASS**
20.00	0.03	0.02%	0.20%	**PASS**
50.00	0.04	0.03%	0.20%	**PASS**
80.00	0.07	0.05%	0.20%	**PASS**
100.00	0.01	0.01%	0.20%	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Performance Qualification

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0
Chromeleon Datasystem	6.80 SP1 Build 2232 (Beta) (Demo-Installation)	Dionex	29461	n.a.

Accessories:

Name	*Description*	*Lot / Serial*	*Exp. Date*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")		
Blank	Water		
Sample 1	Nitrate, 5 ppm		
Sample 2	Nitrate, 10 ppm		
Sample 3	Nitrate, 25 ppm		
Sample 4	Nitrate, 50 ppm		
Sample 5	Nitrate, 100 ppm		
Sample 6	Nitrate, 1000 ppm		
Eluent	Water		
Autosampler Reservoir A	Water		
Balance			
Temperature Probe			
IC Validation Test Box			
Ammeter / Multimeter			

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Customer signature indicates that all information in the following reports has been reviewed and accepted.

Limits:

Test	Customized Limits	Dionex Recommended Limits
ICS-2000 Conductivity Noise (nS)	<= 2.0	<= 2.0
ICS-2000 Conductivity Drift (nS/hr)	<= 20	<= 20
Injector Precision (Area %RSD)	<= 1.0	<= 1.0
Injector Carry Over (Area %)	<= 0.10	<= 0.10
ICS-2000 Detector Linearity (Corr.)	>= 0.999	>= 0.999
ICS-2000 Detector Linearity (%RSD)	<= 5.0	<= 5.0
Eluent Generator Step Current (mA)	See Worksheet	See Worksheet
ICS-2000 Pump Flow Rate Accuracy (mL/min)	<= 0.05	<= 0.05
ICS-2000 Pump Flow Rate Precision (%RSD)	<= 2.0%	<= 2.0%
ICS-2000 Column Heater Temp. Verification (+/- °C)	+/- 2.5	+/- 2.5

Additional Information:

Customer/Company: **Mr. Citizen / XYZ Company**

Qualification Executor/Company: **Mr. Civilian / Dionex Corp.**

Date: **17-May-06**
Period between Qualifications: **6 months**
Next Qualification: **Nov-06**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

DIONEX

Performance Qualification

Detector Noise and Drift

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0

Accessories

Name	*Description*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")	
Eluent	Water	

Additional Information

Customer/Company:	**Mr. Citizen / XYZ Company**	Date:	**17-May-06**
Qualification Executor/Company:	**Mr. Civilian / Dionex Corp.**	Next Qualification:	**Nov-06**

Test Results Summary

Test	**Result**
ICS-2000 Conductivity Noise (nS)	**PASS**
ICS-2000 Conductivity Drift (nS/hr)	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Data for detector noise

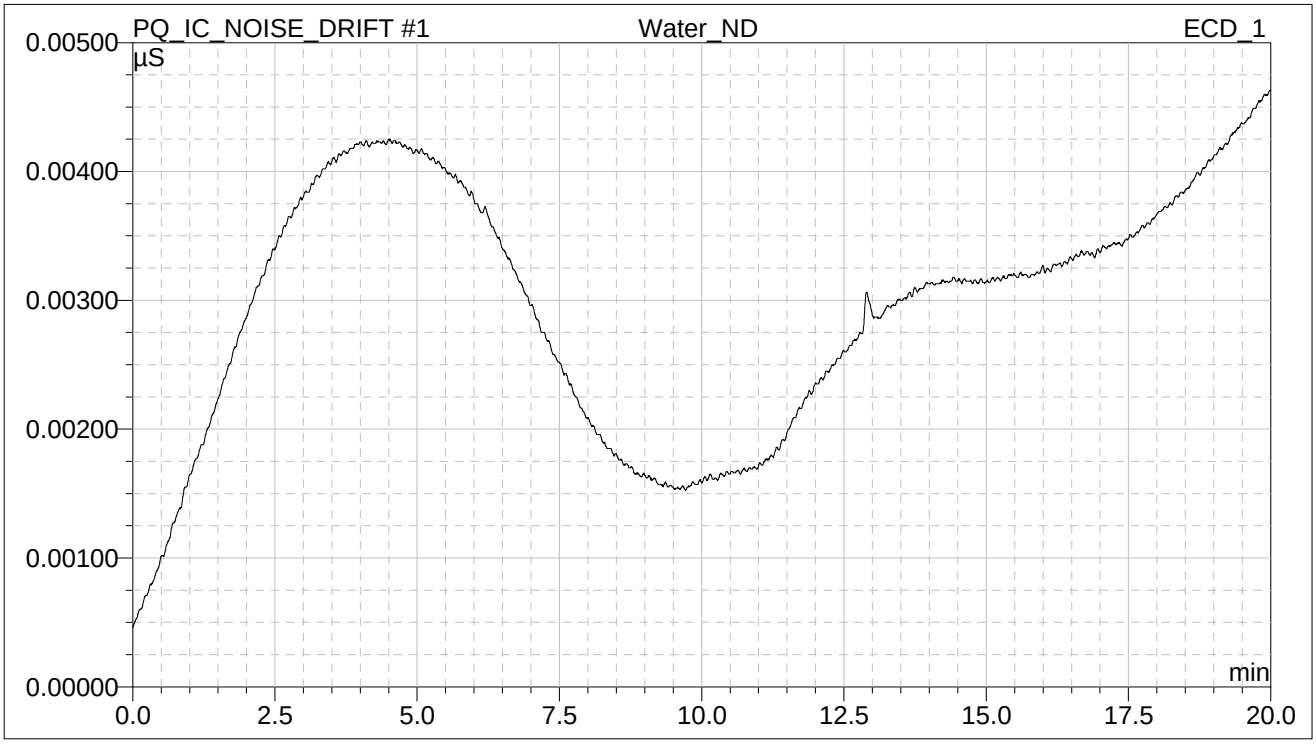
Segment number	Noise, nS
1	0.107
2	0.084
3	0.111
4	0.103
5	0.089
6	0.083
7	0.134
8	0.082
9	0.105
10	0.104
11	0.064
12	0.144
13	0.280
14	0.068
15	0.059
16	0.074
17	0.099
18	0.099
19	0.069
20	0.054
Average, nS	***0.101***
Limit, nS	***2***
Result	***PASS***

Data for detector drift

20 Minute drift, nS	Drift, nS/hr	Limit, nS/hr	Result
0.897	2.691	20.000	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Chromatogram of Detector Noise and Drift



Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Performance Qualification

Injector Precision

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0

Accessories

Name	*Description*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")	
Sample 4	Nitrate, 50 ppm	
Eluent	Water	

Additional Information

Customer/Company:	**Mr. Citizen / XYZ Company**	Date:	**17-May-06**
Qualification Executor/Company:	**Mr. Civilian / Dionex Corp.**	Next Qualification:	**Nov-06**

Test Results Summary

Test	**Result**
Injector Precision (Area %RSD)	**FAIL**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Data for Injector Precision test

Name	***Area µS*min Nitrate ECD_1***
Inj Precision_1	3.027
Inj Precision_2	2.508
Inj Precision_3	2.496
Inj Precision_4	2.526
Inj Precision_5	2.504
Inj Precision_6	2.502
Inj Precision_7	2.509
Inj Precision_8	2.494
Inj Precision_9	2.518
Inj Precision_10	2.501
Average:	***2.558***
Std. Dev:	***0.165***
% RSD:	***6.443 %***
Limit:	***1.0 %***
Result:	***FAIL***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Performance Qualification

Injector Carry Over

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0

Accessories

Name	*Description*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")	
Sample 6	Nitrate, 1000 ppm	
Blank	Water	
Eluent	Water	

Additional Information

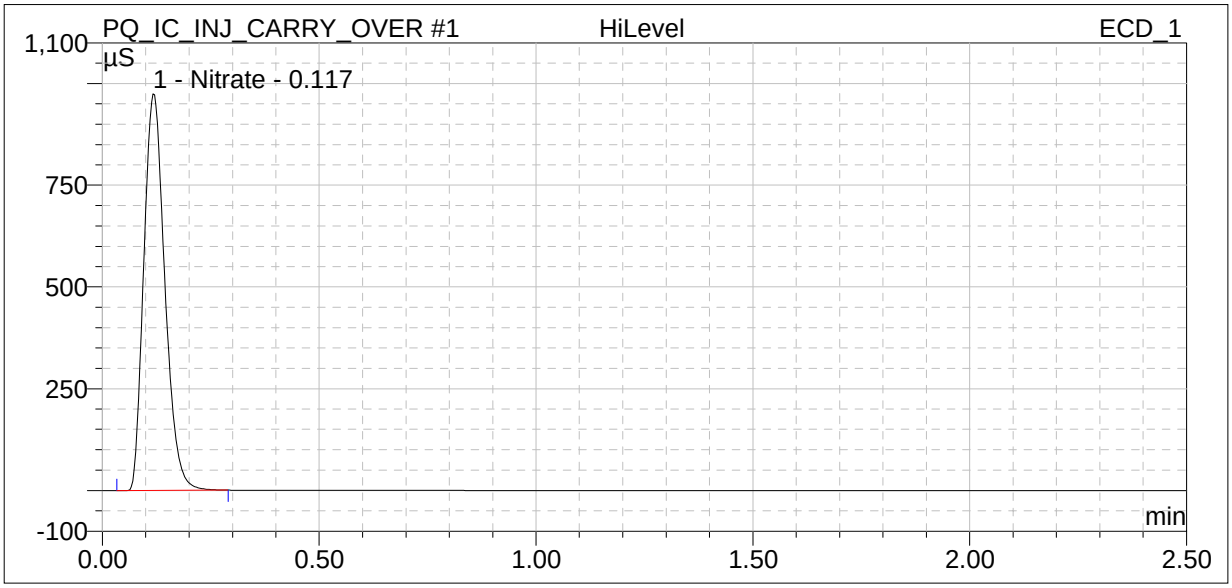
Customer/Company:	**Mr. Citizen / XYZ Company**	Date:	**17-May-06**
Qualification Executor/Company:	**Mr. Civilian / Dionex Corp.**	Next Qualification:	**Nov-06**

Test Results Summary

Test	**Result**
Injector Carry Over (Area %)	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Chromatogram for Carry Over test



Data for Carry Over test

Name	***Ret.Time (detected)*** ***min*** ***Nitrate*** ***ECD_1***	***Area*** ***µS*min*** ***Nitrate*** ***ECD_1***
HiLevel	0.12	54.853
Carry Over	0.12	0.074
Water	0.12	0.043
Carry over:		***0.056 %***
Limit:		***0.100 %***
Result:		***PASS***

DIONEX

Performance Qualification

Detector Linearity

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0

Accessories

Name	*Description*	*Lot / Serial*
Backpressure Tubing	0.13 mm (0.005") ID PEEK, 13 m (512")	
Sample 1	Nitrate, 5 ppm	
Sample 2	Nitrate, 10 ppm	
Sample 3	Nitrate, 25 ppm	
Sample 4	Nitrate, 50 ppm	
Sample 5	Nitrate, 100 ppm	
Eluent	Water	

Additional Information

Customer/Company:	**Mr. Citizen / XYZ Company**	Date:	**17-May-06**
Qualification Executor/Company:	**Mr. Civilian / Dionex Corp.**	Next Qualification:	**Nov-06**

Test Results Summary

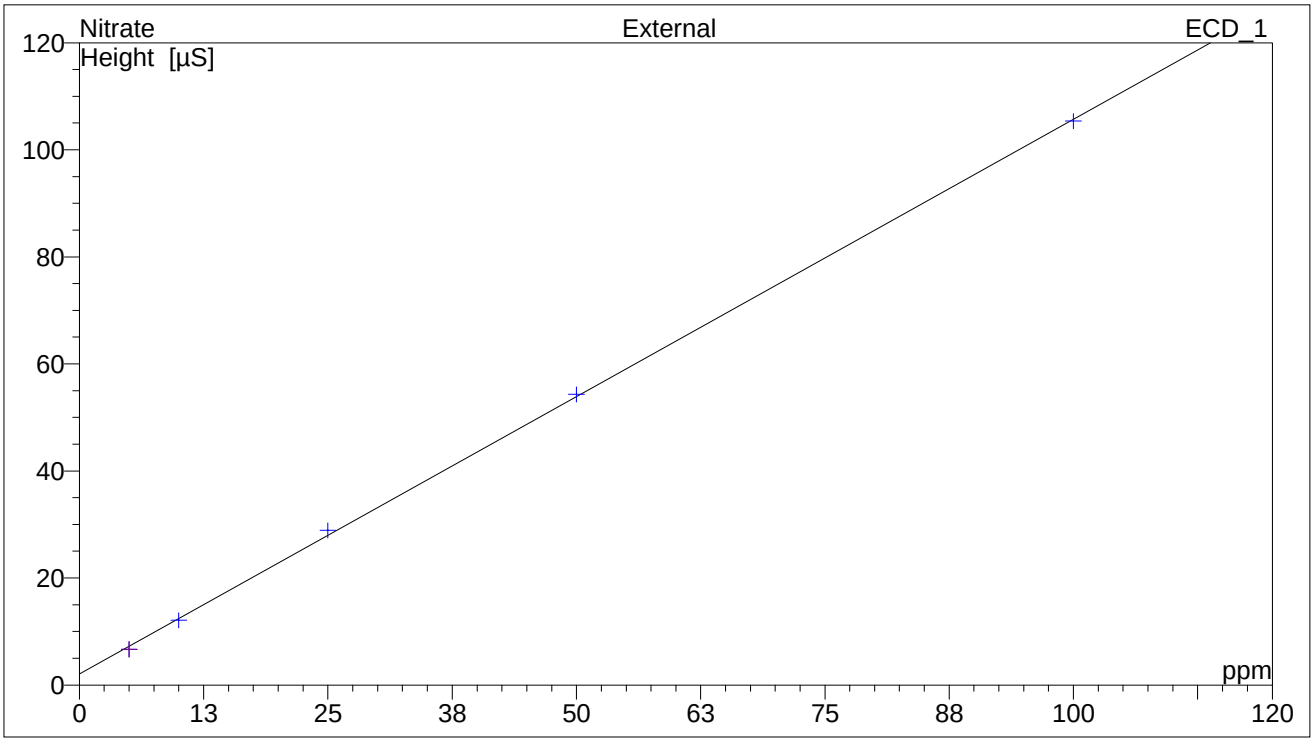
Test	**Result**
ICS-2000 Detector Linearity (Corr.)	**PASS**
ICS-2000 Detector Linearity (%RSD)	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Data for Detector Linearity

Name	Amount ppm Nitrate ECD_1	Height µS Nitrate ECD_1
Detector linearity_1	5.000	6.680
Detector linearity_2	10.000	12.095
Detector linearity_3	25.000	28.884
Detector linearity_4	50.000	54.291
Detector linearity_5	100.000	105.363

Linearity Plot



Calibration Type	Number of Points	Offset	Slope
LOff	5	2.080	1.036

	Correlation Coefficient	% RSD
Linearity:	1.000	1.728
Limit:	**0.999**	**5**
Result:	**PASS**	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Performance Qualification

ICS-2000 Current Qualification

Instruments:

Instrument Name	*Model*	*Supplier*	*Serial Number*	*Moduleware Version*
Pump	ICS-2000	Dionex	05090474	2. 1. 0
Detector	ICS-2000	Dionex	05090474	2. 1. 0
Autosampler	AS40 or man. inj.	Dionex	n.a.	n.a.
Eluent Generator	ICS-2000	Dionex	05090474	2. 1. 0

Accessories

Name	*Description*	*Lot / Serial*
IC Validation Test Box		
Ammeter / Multimeter		

Additional Information

Customer/Company:	**Mr. Citizen / XYZ Company**	Date:	**17-May-06**
Qualification Executor/Company:	**Mr. Civilian / Dionex Corp.**	Next Qualification:	**Nov-06**

Test Results Summary

Test	**Result**
Eluent Generator Step Current (mA)	**PASS**

Customer Signature _____ Date _____ Qualification Executor _____ Date _____

Data for EluGen-MSA Current Qualification Test

Concentration (mM)	*Theoretical Current (mA)*	*Measured Current (mA)*	*Limit (+/-)*	*Deviation*	*Result*
0	0.00	0.00	0.50	0.00	***PASS***
2	3.22	3.21	0.50	-0.01	***PASS***
3	4.82	4.82	0.50	0.00	***PASS***
4	6.43	6.43	0.60	0.00	***PASS***
5	8.04	8.04	0.70	0.00	***PASS***
10	16.08	16.09	0.80	0.01	***PASS***
15	23.60	23.61	0.96	0.01	***PASS***
20	32.02	32.04	1.30	0.02	***PASS***
25	40.94	40.97	1.60	0.03	***PASS***
30	50.08	50.12	1.90	0.04	***PASS***
35	59.47	59.52	2.30	0.05	***PASS***
40	69.09	69.14	2.60	0.05	***PASS***
50	89.03	89.10	3.30	0.07	***PASS***
60	109.91	109.99	4.10	0.08	***PASS***
80	154.48	154.59	6.00	0.11	***PASS***
100	202.80	202.95	8.30	0.15	***PASS***

Customer Signature _____ Date _____ Qualification Executor _____ Date _____